151862

QUALITY ASSURANCE PROJECT PLAN

PRELIMINARY DESIGN INVESTIGATIONS

Northside Sanitary Landfill/
Environmental Conservation
and Chemical Corporation
Indiana

WA 28-5LH2.0
WA 77-5L30.1

August 1987

Remedial Planning Activities
(REM IV) ZONE II
Contract Number 68-01-7251

QUALITY ASSURANCE PROJECT PLAN (QAPP)

Project Title: NSL/ECC, Indiana

EPA Nos.: 28-5LH2.0/77-5L30.1

EPA Remedial Project Officer: Gregg Kulma (acting)

Prepared By: CH2M HILL Date: 8/6/87

Approved _____ Date: 8/6/87
CH2M HILL Site Manager

Approved Karen A. Vendl Date: 8/7/87
EPA Remedial Project Manager

Reviewed _____ Date: 8/7/87
EPA Director, Central Regional Laboratory

Approved James M. Adams, Jr. Date: 8/7/87
EPA QA Officer

GLT718/12

CONTENTS

CONTENTS (continued)

GLT718/14

QUALITY ASSURANCE PROJECT PLAN
NORTHSIDE SANITARY LANDFILL (NSL)/
ENVIRONMENTAL CONSERVATION AND
CHEMICAL CORPORATION (ECC)
PRELIMINARY DESIGN INVESTIGATIONS
INDIANA

INTRODUCTION

The United States Environmental Protection Agency (U.S. EPA) requires participation of all U.S. EPA contractors in a centrally managed quality assurance (QA) program. This requirement applies to all environmental monitoring and measurement efforts mandated or supported by U.S. EPA.

Each contractor generating data has the responsibility to implement minimum procedures to assure that the precision, accuracy, completeness and representativeness of its data are known and documented. To ensure the responsibility is met uniformly, each U.S. EPA contractor must prepare a written Quality Assurance Project Plan (QAPP) covering each project it is contracted to perform.

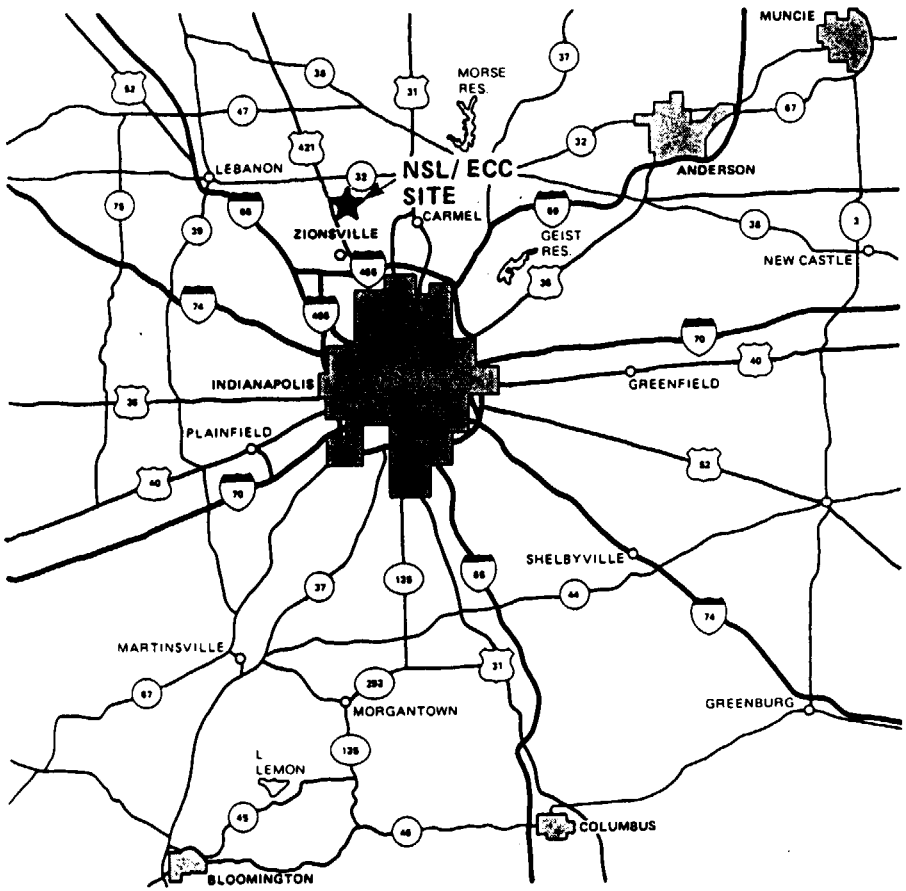
This QAPP presents the organization, objectives, functional activities and specific QA and quality control (QC) activities associated with data collection at the NSL/ECC site near Zionsville, Indiana. This QAPP is designed to achieve the specific data quality goals required for the study plan for collection of preliminary design parameters.

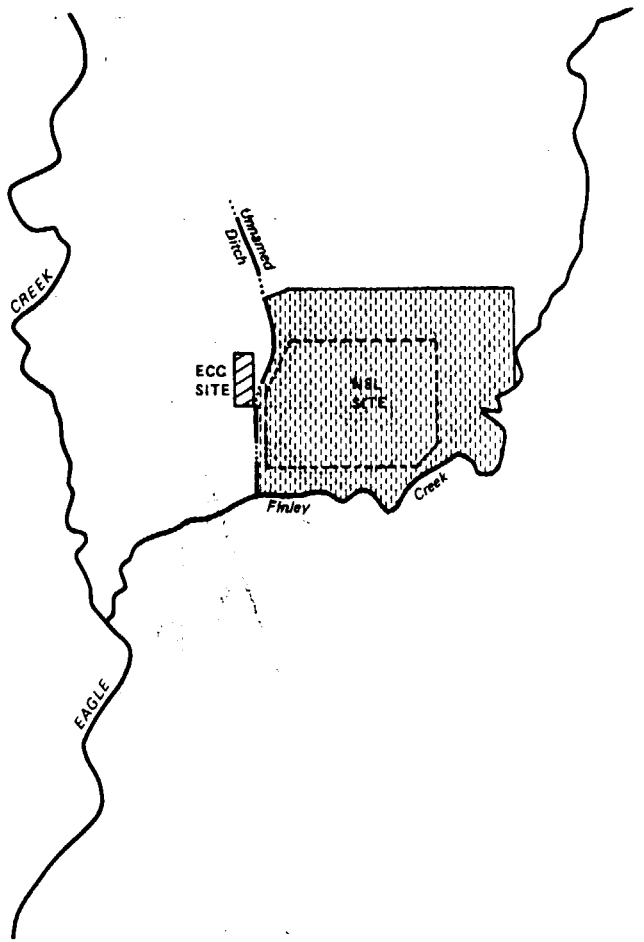
PROJECT DESCRIPTION

This portion of the RI/FS is designed to gather specific information necessary to determine pilot and bench scale test procedures and sizing to support the preparation of contract documents for the collection of predesign data. All tasks and subtasks are directed toward accomplishment of this objective.

BACKGROUND

The ECC and NSL sites are next to each other in a rural area of Boone County, Indiana, south of the intersection of State Route 32 and U.S. Highway 421 and about 10 miles northwest of Indianapolis. The ECC site occupies 6.5 acres immediately west of the 168-acre NSL site, of which approximately 70 acres is landfilled (refer to Figure 1).





LEGEND

NSL SITE

ECC SITE

LANDFILL AREA

SOURCE: U.S.G.S. 7.5 min.quad-rangle, Roseton, Ind. 1969.

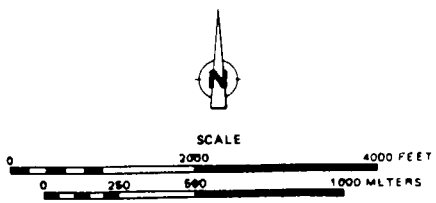


FIGURE 1
LOCATION MAP
NSL/ECC QAPP

NSL is a privately owned and operated active solid waste disposal facility. The site has been active since at least 1962 and has accepted various industrial and municipal wastes during the course of its operation. The vice president of NSL has estimated 16 million gallons of hazardous wastes have been disposed of in the landfill. A 3-acre oil separation lagoon on the landfill surface is evident in a 1977 aerial photograph. The site has had recurring operational deficiencies as reported by the Indiana State Board of Health (ISBH). The U.S. EPA detected leachate running into Finley Creek, and groundwater contamination was detected in monitoring wells at the site. The site was placed on the National Priorities List in 1983.

ECC began operations in 1977 and was engaged in the recovery/reclamation/brokering of primary solvents, oils, and other wastes received from industrial clients. Waste products were received in drums and bulk tankers and prepared for subsequent reclamation or disposal. Reclamation processes included distillation, evaporation, and fractionation to reclaim solvents and oil.

Several memorandums from ISBH discuss the disposal of ECC wastes in the NSL landfill. ECC wastes reportedly disposed of at NSL were 5,000 gallons/month of wastewater from the ECC oil reclamation process, still bottoms and solvent recovery waste, 50 to 80 drums/day of paint sludge, thinner, stain and resin sludge, and at least 7,000 drums of unreported contents.

Drum shipments to ECC were halted in February 1982 after U.S. EPA and ISBH investigations showed accumulation of contaminated stormwater onsite, inadequate management of drum inventory, and several spill incidents. In 1983 ECC was placed on the National Priorities List (NPL) of hazardous waste sites. U.S. EPA subsequently conducted removal actions at ECC including removal, treatment, and disposal of cooling pond waters, about 30,000 drums of waste, 220,000 gallons of hazardous waste from tanks, and 5,650 cubic yards of contaminated soil and cooling pond sludge.

The area surrounding the sites is largely undeveloped. Land use to the east and south of the site is agricultural, to the west and north it is residential. Approximately 50 residences are within 1 mile of the site.

An unnamed drainage ditch that separates NSL from the ECC site flows into Finley Creek near the southwest corner of the landfill. Finley Creek discharges into Eagle Creek about

1/2-mile downstream of the site. Eagle Creek then flows south for about 9 miles before emptying into the Eagle Creek Reservoir, which is used by the City of Indianapolis for a portion of its drinking water supply.

Remedial investigations including soil, hydrogeologic, surface water, and sediment investigations of the sites began in 1983 and continued to November 1985. Details of the investigations are included in the ECC and NSL Remedial Investigation Reports.

Soil contaminants found onsite at the ECC site were primarily volatile organic compounds (VOC's) and phthalates. Migration of VOC's in the soil to the shallow saturated silty clay zone has occurred onsite. The shallow sand and gravel deposit (approximately 18 feet below ground surface) has also been found to be contaminated with VOC's though the source may have been a former cooling pond onsite rather than downward migration from the shallow saturated zone. Organic contaminants were also found in Finley Creek immediately downstream of the site.

Soil contaminants found in peripheral subsurface soils at the NSL site were primarily base/neutral organics and some VOC's at depths of 13 to 15 feet. The sand and gravel lens near the surface in the southwest corner of the site (the lens constitutes the shallow sand and gravel deposit beneath the ECC site) has also been found to be contaminated with VOC's. PAH and VOC contaminants were also found in Finley Creek immediately downstream of the site.

The Feasibility Study Reports for the NSL and ECC sites (dated December 5, 1986) contain more detailed information on the nature of site contamination and site hazards. The recommended alternative to remediate the site includes groundwater and leachate collection and treatment.

PROJECT OBJECTIVES AND DATA USAGE

The objective of this field activity is to gather current data to support the development of the bench and pilot scale treatability tests for inorganic and organic removals from groundwater and leachate. Sizing, procedures and a scope of work is to be defined from resultant data. Data obtained from ISBH will be utilized in sizing the pilot system, thereby assessing the practicality of bench and pilot treatability tests. CLP data will be used to assess the required removal of toxics form the groundwater and leachate prior to discharge. Data accumulated from OVA testing will be used as a means of

monitoring the safety of sampling personnel. Spot test data will be used in the determination of cyanide preservation requirements.

Sampling is proposed to characterize source quantities and strength of leachate being collected in the three onsite leachate tanks and groundwater from five existing wells along the south-southwest perimeter of the site which lie along the general alignment of the proposed groundwater collection system. Approximate quantities must be estimated to determine whether adequate supplies of groundwater and leachate will be available to operate the pilot study during the proposed study period. This will be accomplished through use of the falling-head slug test data (currently available data from the RI) from the wells proposed for use and through gaging the leachate tanks during a 6-day fill cycle to estimate the fill rate of each tank.

The characterization of the influent for the treatment test(s) will be determined through analysis for CLP RAS HSL acid, base/neutral extractables, volatile organics and inorganic compounds including cyanide. Additional analysis will be performed by ISBH for the following conventional parameters: 5-day biochemical oxygen demand, chemical oxygen demand, total organic carbon, total dissolved solids, total suspended solids, volatile suspended solids, chlorides, alkalinity, total phosphorus, sulfate, ammonia nitrogen, total Kjehdal nitrogen, oil and grease, nitrate and nitrite. Field analysis will be performed by the sampling team for the following parameters: pH, conductivity, OVA air monitoring for volatile organic compounds, and a spot test for sulfides. The analyses for HSL compounds will provide information relative to the types of compounds requiring removal as well as information on the types of compounds that may be toxic to treatment biological systems or which may accumulate in the treatment system sludges. The conventional parameter analysis will provide the required information for sizing the pilot biological system based on total organic load and will provide information relative to available nutrients and buffering capacity of the water to define any needs for nutrient addition or pH adjustment. The various measures of organic strength can be used together to estimate any nondegradable or refractory organic fraction that may not be removed by biological oxidation.

Task FT--Source Testing--Groundwater. The samples from groundwater monitoring wells at the NSL/ECC Site will be obtained from five existing wells which lie along the general alignment of the proposed groundwater collection system. Each

well will be sampled once daily for five consecutive sampling days. Static water levels will be recorded before any sampling activity. When practical, three to five casing volumes of water will be purged from each well, using a peristaltic pump or a submersible pump, before collecting samples. Samples will be analyzed for pH, conductivity, and temperature in the field. Both filtered and unfiltered samples will be sent to the U.S. EPA Contract Laboratory Program (CLP) for analysis of the organic (acid and base/neutral extractables, and volatiles) parameters and inorganic (metals and cyanide) constituents as defined in the Users Guide to CLP. Unfiltered monitoring well samples will be analyzed by the Indiana State Board of Health (ISBH) laboratories for biochemical oxygen demand (BOD), chemical oxygen demand (COD), total organic carbon (TOC), total suspended solids (TSS), volatile suspended solids (VSS), total dissolved solids (TDS), kjeldahl nitrogen (TKN), chlorides, sulfates, nitrate/nitrite, alkalinity, ammonia nitrogen, total phosphorous, oil and grease.

Organic samples including acid extractables and base/neutral extractables can be collected using a pump and Teflon tubing or stainless steel bailer. Volatile organics samples will be collected using a Teflon or stainless steel bailer. Metals samples will be field filtered immediately after collection. A detailed description of groundwater sampling procedures is included in the sampling plan (Appendix A). Thirty-one groundwater samples (including replicates and blanks) will be submitted for analysis.

Task FT--Source Testing--Leachate. Samples from the leachate collection tanks at NSL/ECC will be obtained once daily for five consecutive sampling days. The samples from each tank will be of mixed tank contents to represent cumulative composite samples from each vessel. Tanks will be empty initially to determine the fill rate from an empty basis and the tank contents will be circulated to mix accumulated leachate in the tank with any fresh infiltration.

The pumped circulation loop will be routed from the observation pumpout port of the tank to below the water surface level through the tank vent via flexible tubing. Each tank will be gaged every 24 hours to determine the water level in order to estimate the volume of daily infiltration.

Sample analysis for leachate samples will be the same as for groundwater samples as shown in Table B-1 of the sampling plan (Appendix A). Volatile organic samples will be collected from the tanks in order to identify VOC's remaining after the recirculation procedure. It is expected that some light VOC's will be released through pumping but the fraction lost is not

expected to be great since the recycle stream will be drawn and discharged below the water surface level and the mixing is not expected to be turbulent. Daily headspace monitoring for released VOC's using an OVA is recommended prior to sampling each tank to document significant increases in air space VOC concentrations and for sampling personnel safety.

A detailed description of tank testing procedures is provided in the Sampling Plan (Appendix A). Nineteen tank samples (including three duplicates) will be submitted for analysis. Field blanks will be submitted daily.

DATA QUALITY OBJECTIVES

The data quality objectives for this task are as follows:

Engineering quality data are required on conventional parameters and field measurements to determine general treatability test sizing, define general operational conditioning, and identify potential treatment contingencies. Standard methods employed in the field of conventional water and wastewater treatment as outlined in Appendix D will provide the required level of certainty for these parameters. Duplicate and blank samples should be provided at 10 percent or once daily, whichever is greater.

Confirmation quality data are required on priority pollutant analysis to verify previous testing results and establish a potential range of compounds and concentrations to be treated. Since water quality is expected to vary with seasonal influences and with site releases over time, the absolute water quality characterization is not required. Water quality is to be defined for the period of this investigation and should not be used for other evaluational purposes. CLP RAS with no modifications will provide the required level of certainty for this task. Duplicate and blank samples should be provided at 10 percent or once daily, whichever is greater.

Survey quality data are required on all field measurements to indicate obvious differences or changes in water quality, to identify conditions incompatible to the proposed treatment types, to identify the presence or absence of sulfide (for use in selection of the appropriate laboratory technique), and to document gross VOC releases during tank contents mixing for consideration in development of the test plan.

SAMPLING AND ANALYSES

The recommended alternative requires collection and treatment of groundwater and leachate. To better characterize the treatment needs, monitoring wells NSL 12, NSL 10S, NSL 9S, NSL 8SA, and SPB 65 will be sampled once daily for 5 consecutive sampling days. Each well will be purged of three to five casing volumes, if possible, prior to sampling on each day.

Leachate collection tank Nos. 1, 2, and 3 will be pumped dry by the landfill owner prior to monitoring water levels. The water level will be gaged and measured daily in each tank (at the same time each day) to estimate the volume of infiltration into each tank for 5 consecutive sampling days.

Analysis of samples is as follows:

- Field determination for pH, conductivity, temperature, spot test for sulfides, and OVA air monitoring for volatile organic compounds.
- CLP RAS analysis for volatile, acid extractable, and base/neutral organics.
- CLP RAS analysis for metals and cyanide (or SAS analysis for cyanide in the presence of chlorine or sulfide).
- ISBH analysis for BOD, COD, TOC, TSS, VSS, TDS, NO_3 + NO_2, sulfate, alkalinity, NH_3, TKN, P, chlorides, oil, and grease.

Procedures are included in appendixes to this QAPP for field determinations, CLP special analytical services, and ISBH analyses. Detail regarding sample collection volumes, preservatives, and containers is included in Appendix A, Sampling Plan, in Attachments B and C. A summary of all anticipated sampling and analyses at the NSL/ECC site are listed in Table B-1 in Appendix A.

The compounds to be determined during the remedial investigation are listed in Attachment A to the Sampling Plan (Appendix A). Computer assisted library searches will also be made to tentatively identify as many as 30 additional organic compounds.

Sampling Schedule

The proposed fieldwork is scheduled to take place the week of August 23, 1987. Samples will be collected from August 24 through August 28. Figure 1A shows the proposed schedule and duration of the proposed tasks.

PROJECT ORGANIZATION AND RESPONSIBILITY

CH2M HILL has overall responsibility for all phases of the RI/FS. CH2M HILL will perform the field investigations and prepare the RI report. Subsequently, CH2M HILL will prepare the Feasibility Study.

Task PM--Project Management. Project management activities will be handled through CH2M HILL's office in Milwaukee, Wisconsin. Contact will be maintained with the U.S. EPA Remedial Project Manager (RPM) during all phases of the project.

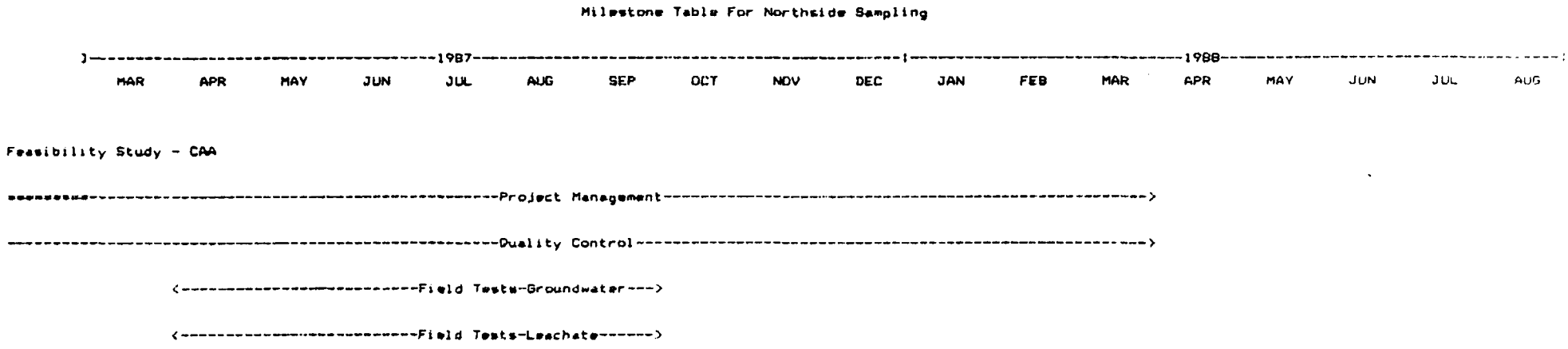
Project management activities will include preparation of monthly reports to keep the U.S. EPA informed of the technical, financial, and schedule status of the project. Other responsibilities include controlling budgets and schedules; selecting, coordinating, and scheduling staff and subcontractors for task assignments; maintaining project quality control and assurance programs.

Task QC--Quality Control. Periodic review of project files, project deliverables, and site inspection during the Field Activities will be conducted by a review team throughout the project. The team will consist of three professionals with experience from appropriate disciplines related to the problems and investigations at the site.

The following responsibilities have been assigned for the project:

- Remedial Project Manager (RPM)
  Karen Vendl (U.S. EPA)
- Site Manager (SM)
  Alpheus Sloan III (CH2M HILL)
- Regional Manager (RM)
  Mike Jury (CH2M HILL)
- Quality Assurance Manager (QAM)
  John Ramage (CH2M HILL)

FIGURE #1A


Milestone Table For Northside Sampling
1987
1988
MAR
APR
MAY
JUN
JUL
AUG
SEP
OCT
NOV
DEC
JAN
FEB
MAR
APR
MAY
JUN
JUL
AUG
Feasibility Study - CAA
Project Management
Quality Control
Field Tests-Groundwater
Field Tests-Leachate

- o CH2M HILL Review Team Leader (RTL)
  Jim Kennedy (CH2M HILL)

- o Sample Team Leader
  Jeff Keiser and CH2M HILL, B&V, and PRC Staff

- o Laboratory Operation
  All samples will be sent to the U.S. EPA Contract Lab Program (CLP) and the Indiana State Board of Health (ISBH).

- o System/Performance Audits
  CH2M HILL QA Manager (field), U.S. EPA EMSL--Las Vegas (CLP RAS), Contract Project Management Section (CPMS), CRL (ISBH).

- o Special Analytical Services Requests Preparation
  CH2M HILL

- o Review of Tentatively Identified Compounds
  CH2M HILL

- o QA/QC of CLP Data--U.S. EPA Region V, Contract Project Management Section (CRL)

- o QA/QC of SAS Data--U.S. EPA Region V, Contract Project Management Section (CRL)

- o CLP Data Completeness--CH2M HILL

- o QA/QC of Indiana State Board of Health Data--U.S. EPA Region V Contract Project Management Section (CRL)

Primary responsibility for project quality rests with the SM. Independent quality assurance review is provided by the QA reviewers. A project organization chart is presented in Figure 2.

QUALITY ASSURANCE OBJECTIVES

The overall QA objectives is to develop and implement procedures for field sampling, chain of custody, laboratory analysis, and reporting that will provide data to evaluate treatment schemes, confirm the nature and extent of contaminants established during the RI/FS, and generate data that is defensible in a court of law for cost recovery purposes. Specific procedures to be used for sampling, chain of custody, calibration, laboratory analysis, reporting, internal quality

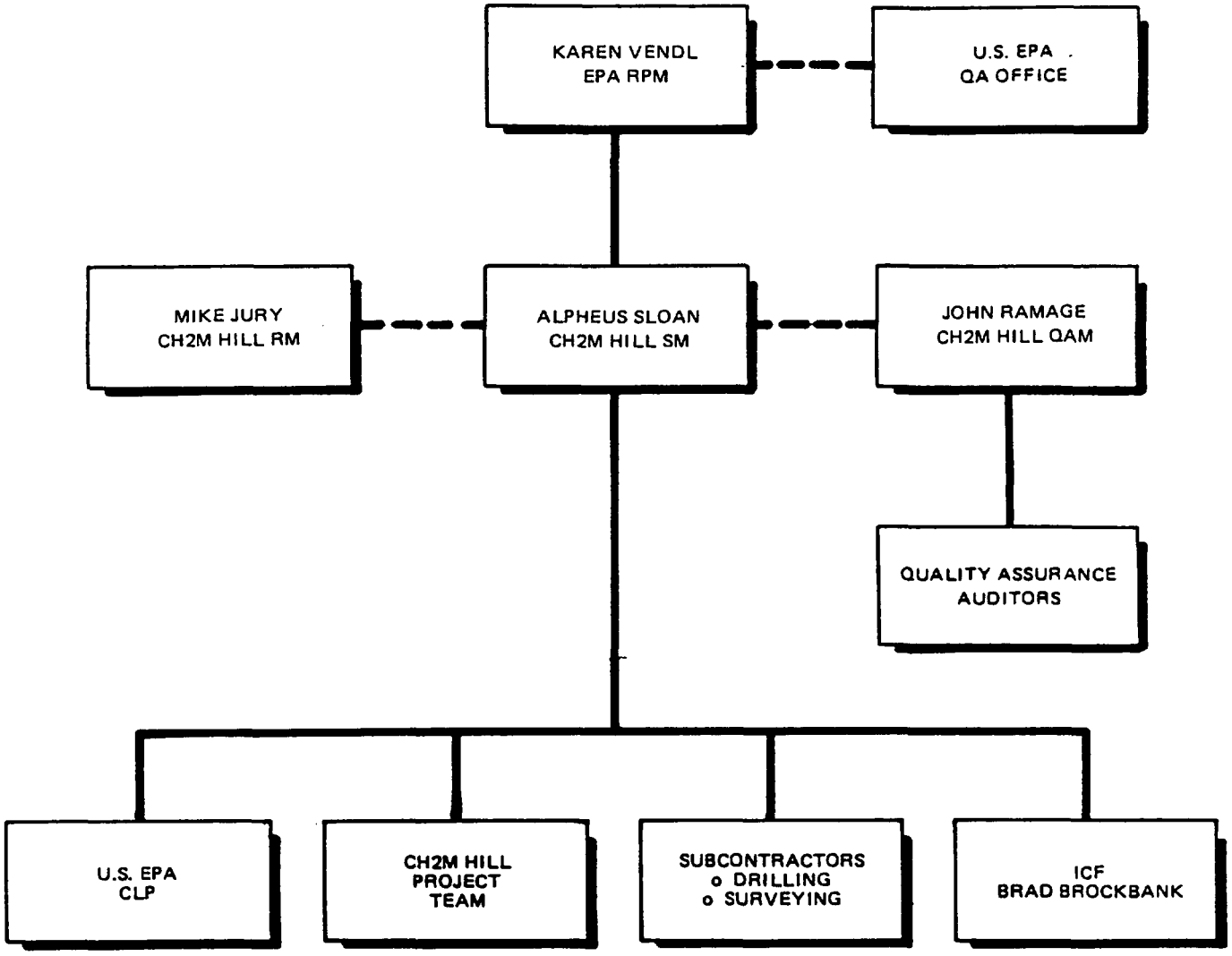


FIGURE 2
PRELIMINARY DESIGN
PROJECT ORGANIZATION
NSL/ECC

control, and its preventative maintenance and corrective actions are described in other sections of this Quality Assurance Project Plan.

To assess the quality of data from field sampling efforts, replicate and blank samples will be submitted. Blank samples will be analyzed to check for procedural contamination and/or ambient conditions at the site which are causing sample contamination.

In Table B-1, a list of replicate and blank groundwater and leachate samples is provided.

ACCURACY, PRECISION, AND SENSITIVITY OF LABORATORY ANALYSIS

All groundwater taken at the NSL/ECC site will be analyzed using the U.S. EPA Contract Laboratory Program (CLP) and the Indiana State Board of Health (ISBH). The QA goals for these analyses are established under CLP guidelines as stated in IFB's WA-85-J664/J680 for organics and WP-85-J838/J839 for inorganics. In addition to routine organic and inorganic analyses, CLP Special Analytical Services (SAS) will analyze groundwater and leachate samples for additional parameters. These parameters and their respective QA objectives are listed in Appendix D.

SAMPLING PROCEDURES

Detailed sampling procedures are provided in the Sampling Plan, Appendix A.

CALIBRATION PROCEDURES AND FREQUENCY

Specific operating and calibration procedures for the pH and specific conductivity meters to be used in the field are contained in Appendixes E, F and G, respectively.

SAMPLE CUSTODY

Sample custody procedures for this project will be in accordance with the procedures detailed in Section 5 of the CH2M HILL REM/FIT Quality Assurance Manual, Sample Control, and the Draft REM/FIT Documentation Protocol for Region V (May 1984).

ANALYTICAL SERVICES

All samples will be analyzed using RAS or SAS of the CLP for priority pollutant and ISBH analyses for conventional parameters (Appendix A). In addition, several field measurements will be performed. QAPP elements for each are listed below and are documented in the references cited.

QAPP Element	RAS	SAS	ISBH	Field Analysis
Calibration Procedures	PD	Appendix C	Appendix D	Appendixes E-G
Analytical Procedures	PD	Appendix C	Appendix D	Appendixes E-G
Internal QC	PD	Appendix C	Appendix D	Appendixes E-G
Data Reduction/Validation	PD	Appendix C	Appendix D	Appendixes E-G
Performance/System Audit	PD	Appendix C	Appendix D	Appendixes E-G
Data Assessment	PD	Appendix C	Appendix D	Appendixes E-G
Accuracy/Precision Definitions	PD	Appendix C	Appendix D	Appendixes E-G
Corrective Action	PD	Appendix C	Appendix D	Appendixes E-G

PD = Predetermined in CLP, IFB Nos. WA-85-J644/J680 for organic chemical analyses and IFB Nos. WP-85-J838/J839 for metals and cyanide.

CLP ROUTINE ANALYTICAL SERVICES

Sample Custody

Chain of custody forms, traffic reports and sample tags will be filled out as samples are collected. These forms are placed in the cooler along with the samples. The cooler is sealed with custody seals.

Upon receipt, the laboratory sample custodian will sign the chain of custody and maintain custody of the samples during analysis. When samples are not in the physical presence of the sample custodian they will remain in a locked and secured area under his control.

After sample analysis has been completed the originals of all paperwork associated with sample custody and tracking will be turned over to National Enforcement and Investigations Center. A complete description of the custody procedures used may be found in the NEIC Policies and Procedures manual revised June 1985.

Analytical and Calibration Procedures

All samples collected will be analyzed for Hazardous Substances List Organics (VOA's, acid, base/neutral extractables) and metals and cyanide by the CLP. All testing of soil, groundwater, surface water, and leachate samples will conform to the guidelines in the User's Guide to the U.S. EPA Contract Laboratory Program and to those specified in IFB's WA-85-J664/J680 for organics and WA-85-J838/J839 for metals and cyanide.

Computer-assisted library searches will be made to tentatively identify as many as 30 organic compounds in addition to those listed in the Sampling Plan (Appendix A). However, no more than 4 hours per sample will be spent in the search for the identity of unknowns. The three most matched compounds will be reported via a computer mass spectral library search. Positive peak identification requires at least a five major-peak match including the base peak and molecular ion peak. The relative intensities of these peaks should not vary by ± 20 percent compared to the suspected compound. Compounds still unidentified after 4 hours are labeled as UNKNOWN #XXX, where XXX is the scan number where the unknown appears.

Internal Quality Control

Internal quality control procedures for groundwater and leachate samples will follow the guidelines of the CLP specified in the IFB's WA-85-J664/J680 for organics and WP-85-J838/J839 for metals and cyanide. Field blanks and replicates will be collected to check for any sample contamination resulting from field sampling equipment and to check data precision, respectively.

Data Reduction, Validation, and Reporting

Data validation will be performed by the CPM Section and the CRL QA Coordinator. The raw data collected from project sampling tasks and used in project reports will be appropriately identified and will be included in a separate appendix within the final report. Where test data have been reduced, the method of reduction will be described. CH2M HILL will perform all data reduction. Any method used for data reduction will be described and be part of the data package.

Performance and System Audit

Performance and systems audits for CLP, RAS, are the responsibility of the Support Services Branch, OERR, U.S. EPA and of EMSL--Las Vegas, U.S. EPA.

The Quality Assurance Manager (QAM) will monitor and audit performance of the QA procedures to assure that the project is performed in accordance with approved quality assurance procedures. The QAM will conduct the audits as described in Section 9, Audit Program, of the CH2M HILL REM/FIT Quality Assurance Manual. Audits may be scheduled at various times to evaluate the execution of sample identification, sample control, chain-of-custody procedures, field notebooks and sampling procedures.

Data Assessment

Data assessment is the responsibility of CPMS, CRL. Data completeness will be checked by CH2M HILL and the SMO.

Accuracy and Precision Definitions

Accuracy and precision definitions for analyses performed by CLP, RAS, are listed in IFB No.'s WA-85-J664/J680 and WP-85-J838/J839.

Corrective Actions

If quality control audits result in detection of unacceptable conditions the laboratory will contact Program Coordinator of the CPM section of the CRL. The project manager and site project manager will be informed of the unacceptable conditions and along with the CPM's will develop and initiate the appropriate corrective action.

SPECIAL ANALYTICAL SERVICES

Sample Custody

Chain of custody forms, traffic reports, and sample tags will be filled out as samples are collected. These forms are placed in the cooler along with the samples. The cooler is sealed with custody seals.

Upon receipt, the laboratory sample custodian will sign the chain of custody and maintain custody of the samples during analysis. When samples are not in the physical presence of

the sample custodian they will remain in a locked and secured area under his control.

After sample analysis has been completed the originals of all paperwork associated with sample custody and tracking will be turned over to National Enforcement and Investigations Center. A complete description of the custody procedures used may be found in the PEIC Policies and Procedures manual revised June 1985.

Analytical and Calibration Procedures

The unfiltered groundwater and leachate samples will be analyzed by the Indiana State Board of Health lab for BOD_5, COD, and TOC. These samples will also be analyzed for TSS, TDS, VSS, chlorides, sulfates, nitrate and nitrite, alkalinity, ammonia nitrogen, total kjeldahl nitrogen (TKN), total phosphorous, chemical oxygen demand (COD), biochemical oxygen demand (BOD), volatile suspended solids (VSS), oil and grease, and total organic carbon (TOC) by the ISBH. Analytical procedures for these analyses are specified in ISBH Special Analytical Services (Appendix D).

Internal Quality Control

Quality control requirements for each of the SAS analyses are specified in Appendixes C and D. Field blanks and duplicates will be collected and submitted for analysis to determine if any sample contamination is due to field sampling equipment and to check data precision, respectively. Field blanks and duplicates are noted on Attachment B-1 to the sampling plan (Appendix A).

Data Reduction, Validation, and Reporting

The test procedures used by SAS will be clearly identified. Bench records and all records of analyses and calculations for samples, blanks, duplicates, spikes, standards, etc., with resulting instrument inputs or concentration readouts, will be provided by CLP, SAS, along with worksheets used to calculate results. The Contract Project Management section of the CRL will perform data validation. The raw data collected and used in project reports will be appropriately identified and included in a separate appendix in the final report. Any method used for data reduction will be described and will be part of the data package.

Performance and System Audit

System audits and required performance limits are specified for each SAS analysis in Appendixes C and D.

Data Assessment

Data Assessment is the responsibility of CPMS, CRL. Data completeness will be checked by CH2M HILL and the SMO.

Accuracy and Precision Definitions

Accuracy and precision definitions are specified for each SAS analysis in Appendixes C and D.

Corrective Actions

If quality control audits result in detection of unacceptable conditions the laboratory will contact Program Coordinator of the CPM section of the CRL. The project manager and site project manager will be informed of the unacceptable conditions and along with the CPM's will develop and initiate the appropriate corrective action.

INDIANA STATE BOARD OF HEALTH LABORATORY ANALYSIS

Sample Custody. Chain of custody and sample tags will be completed in the field as samples are collected. Chain of custody forms will be shipped in coolers with the samples. All coolers will be shipped with custody seals attached at opposite corners. The sample custodian at the laboratory will sign the chain of custody form upon receipt of the samples. Samples will remain in the custody of the sample custodian until completion of the analysis. All sample tags and completed chain of custody forms will be returned to CH2M HILL upon completion of the analysis. CH2M HILL will maintain the final evidence files until the project is complete. At that time the evidence files will be turned over the U.S. EPA along with the rest of the project files.

Analytical and Calibration Procedures. Special Analytical Services Request Forms have been filled out with ISBH procedures attached for BOD_5, COD, TOC, TSS, VSS, TPS, Nitrate, Nitrite, TKN, ammonia, total phosphorus, alkalinity, chlorides and sulfates. This was done to ensure complete documentation of analysis and quality control.

Internal Quality Control. Quality control requirements for each of the ISBH analyses are specified in Appendix D. Field blanks and duplicates will be collected and submitted to ISBH for analysis. These samples will be used to determine if any contamination is due to field sampling and to check precision.

Data Reduction, Validation, and Reporting. The test procedures used are clearly identified in Appendix D. Bench records and all records of analyses and calculations for samples, blanks, duplicates, spikes, standards, etc., with resulting instrument readouts will be provided along with worksheets used to calculate results. The raw data collected and used in project reports will be appropriately identified and included in a separate appendix in the task TM. Any method used for data reduction will be clearly described and will be included as part of the data package.

Performance and System Audits. Performance and systems audits for ISBH services are the responsibility of the Contract Project Management Section (CPMS), CRL, Region V, U.S. EPA.

Systems audits and required performance limits are specified for each ISBH analysis in Appendix D.

The Quality Assurance Manager (QAM) will monitor and audit performance of the QA procedures to assure that the project is performed in accordance with approved quality assurance procedures. The QAM will conduct the audits as described in Section 9, Audit Program, of the CH2M HILL REM/FIT Quality Assurance Manual. Audits may be scheduled at various times to evaluate the execution of sample identification, sample control, chain-of-custody procedures, field notebooks, and sampling procedures.

Data Assessment. Data assessment will be the responsibility of CPMS, CRL. Data completeness will be checked by CH2M HILL.

Accuracy and Precision Definitions. Accuracy and precision are specified for each ISBH analysis in Appendix D.

Corrective Actions. If quality control audits detect unacceptable conditions or data, samples should be reanalyzed if holding time criteria permit. CH2M HILL should be contacted if requirements are not met upon reanalysis of samples.

FIELD ANALYSES

Analytical and Calibration Procedures

Groundwater, surface water, and leachate samples will be analyzed for pH, specific conductivity, and temperature. A spot test for sulfides will be performed in the field for samples intended for cyanide analysis. Analytical and calibration procedures for pH determinations are given in Appendix E and those for specific conductivity and temperature in

Appendix F. Appendix G contains the spot test procedure for sulfide.

Internal Quality Control

Field analyses are performed onsite and do not involve samples that are collected and retained. The primary QA/QC objective is to obtain reproducible measurements to a degree of accuracy consistent with limits imposed by analytical methodologies used and with the intended use of the data. Quality control procedures will be limited to checking the reproducibility of measurements by taking multiple readings and by calibration of instruments (where appropriate).

Data Reduction, Validation, and Reporting

All field recording sheets, instrument outputs, and worksheets for calculating results will be retained. Summarized raw data will be appropriately identified in reports and included in a separate appendix of the final report.

Performance and System Audit

All instruments used in making field measurements will be regularly calibrated (where appropriate) as specified in Appendixes E through G.

Data Assessment

The Quality Assurance Manager (QAM) will assess data to assure QA/QC objectives are met.

Accuracy and Precision Definitions

No quantitative levels are specified.

Corrective Actions

If variability among multiple readings at a single site is judged excessive, instruments will be recalibrated (if appropriate) and the measurement repeated. If variability remains unacceptably high and instruments fail to properly calibrate, the QAM will be notified.

QUALITY ASSURANCE REPORTS

No separate QA report for this project is anticipated. The final report will contain separate QA sections that summarize data quality information collected during the project.

GLT718/15

Appendix A
SAMPLING PLAN

CONTENTS

GLT718/5

SAMPLING PLAN
NORTHSIDE SANITARY LANDFILL (NSL)/ ENVIRONMENTAL CONSERVATION AND CHEMICAL CORPORATION (ECC) PRELIMINARY DESIGN INVESTIGATIONS INDIANA

OBJECTIVE

This sampling plan documents procedures and practices to be used in obtaining samples of groundwater and leachate at the site. Five wells and three leachate tanks will be sampled over a 5 day period.

SAMPLE LOCATIONS AND NUMBERS

SAMPLE LOCATIONS

Groundwater Samples

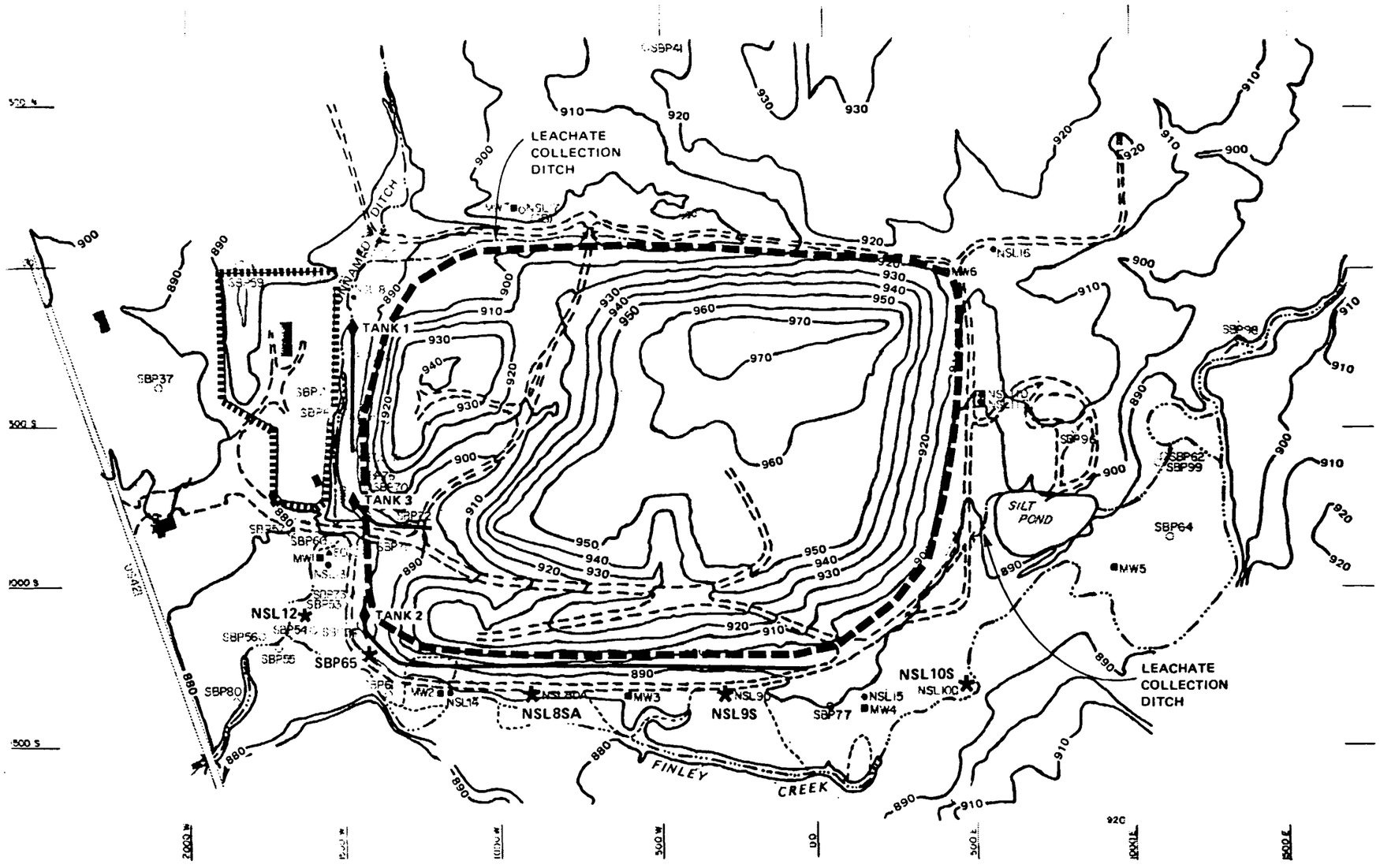
Groundwater samples will be collected from five existing monitoring wells onsite which lie along the general alignment of the proposed groundwater collection system. Figure A-1 shows the approximate location of the monitoring wells to be sampled. Replicates ultra-pure water blanks and field blanks will be taken as specified on Attachment B-1. The blanks and replicate samples will be preserved in the same manner as the other groundwater samples. The field blank will be bottled using the sampling equipment as a check to measure field decon and sampling interferences or influences.

Leachate Samples

Leachate samples will be collected from the three onsite buried leachate collection tanks. Figure A-1 shows the approximate location of the tanks to be sampled. Blanks and replicates are specified in Attachment B-1 in conjunction with the groundwater samples.

SAMPLE DESIGNATION

All samples regardless of destination, will carry a CH2M HILL number which indicates origin of sample (i.e., groundwater). Samples sent to CLP will carry the CRL number while ISBH samples will carry traveling numbers, but not CRL numbers. Other identification of samples include numerical designators assigned to samples to correspond to tracking documentation as described in Appendix I. A Sample Management Office (SMO)



LEGEND

- ~900~ CONTOUR LINES
- LANDFILL ROADWAYS
- CREEKS
- FORMER CREEK AND DITCH BED
- DRAINAGE DITCH
- ♦ BURIED LEACHATE COLLECTION TANK TO BE SAMPLED
- BURIED LEACHATE TILES (APPROXIMATE LOCATION)
- ECC SITE BOUNDARY
- APPROXIMATE BOUNDARY OF NSL LANDFILLED AREA
- NSL10S ✱ MONITORING WELL TO BE SAMPLED

NOTES: This map is based on a 1983 topographic map from Harding Lawson Associates. Soil excavation has occurred on the northeast since that time. Former Finley Creek location based on maps from M.E. AYDT, P.E., 08-22-79 and the Indiana State Board of Health.

N

0 500

250

SCALE IN FEET

FIGURE A-1
SITE FEATURES
ECC - NSL QAPP

number and a Central Region Lab (CRL) number will be assigned to each sample at the same time. Refer to the user's guide to the CLP for an explanation of the SMO numbers. A listing of sample identification numbers will be maintained in the log book by the Team Leader. Each CH2M HILL sample number will consist of three components as described below:

Project Identification

A three-letter designation will be used to identify the site where the sample was collected. For this project it will be NSL for Northside Landfill.

Sample Location

Each sample collected will be identified by an alpha-code corresponding to the sample type, followed by the sample location number. The alpha-codes are as follows:

- LT--Leachate Tank
- MW--monitoring well, groundwater

Field blanks will have an FB followed by the alpha code for the type of blank (i.e., a surface water blank will be FBSW).

Sample Identifier

All samples will have a two-digit number as the last component of the sample identifier. The sampling events will start with 01 and progress upward.

Sample Number Examples

NSL-MW01-02 Northside Landfill--groundwater sample 2 from monitoring well MW01

NSL-LT02-01 Northside Landfill--leachate tank sample 1 from location LT02

SAMPLING EQUIPMENT AND PROCEDURES

GROUNDWATER SAMPLE COLLECTION

Prior to purging each well for sampling, a water level measurement will be taken using an electronic device with a stainless steel probe. A horn sounds when the probe makes contact with the water surface. The device will then be used to measure the total depth of the well to verify well

identification. All water purged from the wells will be released onsite.

Each well to be sampled will be purged immediately prior to sampling using either a stainless steel or Teflon bailer, a submersible positive displacement pump (Johnson Keck), or a peristaltic pump. Discharge water will be collected and measured so that three to five well volumes are removed prior to sample collection. If pumps are used, the bottom 5 feet of hose will be Teflon so the hose will not contaminate the well or well water. If the well does not recharge, the well will be bailed dry and allowed to recharge over 24 hours.

After the well has been purged, the samples will be collected using a stainless steel or Teflon bottom loading bailer. Before samples are collected, approximately one-half of the well water volume will be removed with a stainless steel bailer. From the remaining well volume, samples will be collected. The bailers will be raised and lowered on a thin stainless steel cable.

All sampling equipment will be cleaned between wells by scrubbing with a trisodium phosphate (TSP) decontamination fluid followed by a 10 percent (by volume) reagent grade methanol mix with distilled water and finally a triple rinsed with distilled water. The TSP decontamination fluid will be tap water with approximately 2.5 percent TSP dissolved (by weight). Sampling equipment will be triple rinsed with distilled water poured directly from the distilled water containers to eliminate methanol contamination. The pump and/or bailers will be laid out on clean plastic to air dry before reuse.

LEACHATE SAMPLE COLLECTION

Prior to setting up each tank for sampling, the tanks will be pumped out by the owner as is currently being done for periodic disposal of tank contents. A water level measurement will be taken using a gaging rod and the depth of the tank contents (initially empty) will be used to determine the daily volume of infiltration into each tank.

Each tank will be equipped with a submersible pump (sump pump type) and ample tubing to recirculate the tank contents from the observation/pumpout port to below the water surface level through the tank vent. The discharge tubing will be weighted at the end to remain submerged and minimize the transfer of VOC's through open air discharge. The

recirculation rate will be set at about twice the estimated fill rate of each tank to provide adequate mixing without excessive turbulence.

Pumps will be dedicated to each tank and will be equipped with tygon flexible tubing for recirculation and sampling. Samples will be drawn using the recirculation system via a coupling provided on the discharge tubing outside the tank. VOC samples will be drawn from the observation/pumpout port of each tank using a stainless steel or teflon bailer. Decontamination of the bailer will be as described for groundwater sampling.

SAMPLE HANDLING AND ANALYSIS

PARAMETERS

Below is a listing of analyses to be conducted on the various sample types collected:

- o Groundwater and Leachate
  - Routine Inorganic Analyses (Metals and Cyanide)--U.S. EPA CLP
  - Routine Organic Analyses (VOC's and base/neutral and acid organics)--U.S. EPA CLP
  - BOD_5, COD, TOC--most NO_3+, NO_2, TSS, TDS, VSS, alkalinity total phosphorus, ammonia, TKN chlorides and sulfides by the ISBH lab
  - pH, temperature, Specific Conductivity (Field measurement)
  - Spot test for sulfides (Field measurement)
  - HNu or OVA organic vapor concentration (Field measurement)

All samples will be considered low concentration samples. The determination of low concentration is based on existing analytical data collected from the site. Routine organic and inorganic parameters are given in Attachment A.

SAMPLE PREPARATION

All samples collected will immediately be placed on ice (if necessary to maintain a temperature of 4°C). Three metals

samples will be collected at each sampling location. These include:

- One sample which will be filtered in the field directly after collection. The sample will be filtered through 0.45 micron filter paper using a pressure filtration device as described in Appendix H.
- The second sample will not be filtered in the field or laboratory.
- A sulfide spot test will be run in the field on the third sample. This procedure is described in Appendix G.

All sample fractions will be preserved prior to shipment according to the following procedures (see Attachment B for greater detail):

- Metals
  - Filtered through 0.45 micron filter (leachate samples will not be filtered)
  - Nitric Acid; to pH less than 2
- Cyanide; NaOH to a pH greater than 12

All samples will be shipped to the contract laboratories and the ISBH laboratories the same day they are collected by overnight express. Attachment B describes shipping methods in greater detail.

SAMPLE DOCUMENTATION

All samples will be collected under chain-of-custody procedures. Standard paperwork including sample tags, traffic reports, chain-of-custody forms, and custody seals used for CLP sample tracking and records will be filled out as described in Appendix I. All pertinent information about the samples will be logged in the site log maintained by the Team Leader. This information will include sample time, location, tag numbers, designation, and sampler. New readings, weather conditions, and field modifications or decisions will also be recorded. The log book will be filled in ink unless weather conditions dictate otherwise. Photographs with the time, date, location, and task description will also be noted in the log book.

WASTE DISPOSAL

During sampling, purge water that registers HNu or OVA readings will be held in Department of Transportation (DOT) approved 55-gallon drums. The full drums will be labeled and stored in a secure area onsite for later disposal, if deemed necessary by U.S. EPA. All protective clothing and sampling-related wastes generated during the activity (i.e., decon solutions) will be disposed of in DOT approved 55-gallon drums.

GLT718/16

Attachment A
ROUTINE ANALYTICAL SERVICES PARAMETERS

Hazardous Substance List (HSL) and Contract Required Detection Limits (CRDL)**

Volatiles	CAS Number	Detection Limits* Low Water[a] ug/L	Low Soil/Sediment[b] ug/Kg
1. Chloromethane	74-87-3	10	10
2. Bromomethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	1	1
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	5	5
6. Acetone	67-64-1	10	10
7. Carbon Disulfide	75-15-0	5	5
8. 1,1-Dichloroethene	75-35-4	5	5
9. 1,1-Dichloroethane	75-35-3	5	5
10. trans-1,2-Dichloroethene	156-60-5	5	5
11. Chloroform	67-66-3	5	5
12. 1,2-Dichloroethane	107-06-2	5	5
13. 2-Butanone	78-93-3	10	10
14. 1,1,1-Trichloroethane	71-55-6	5	5
15. Carbon Tetrachloride	56-23-5	5	5
16. Vinyl Acetate	108-05-4	10	10
17. Bromodichloromethane	75-27-4	5	5
18. 1,1,2,2-Tetrachloroethane	79-34-5	5	5
19. 1,2-Dichloropropane	78-87-5	5	5
20. trans-1,3-Dichloropropene	10061-02-6	5	5
21. Trichloroethene	79-01-6	5	5
22. Dibromochloromethane	124-48-1	5	5
23. 1,1,2-Trichloroethane	79-00-5	5	5
24. Benzene	71-43-2	5	5
25. cis-1,3-Dichloropropene	10061-01-5	5	5

(continued)

Volatiles	CAS Number	Detection Limits* Low Water[a] ug/L	Low Soil/Sediment[b] ug/Kg
26. 2-Chloroethyl Vinyl Ether	110-75-8	10	10
27. Bromoform	75-25-2	5	5
28. 2-Hexanone	591-78-6	10	10
29. 4-Methyl-2-pentanone	108-10-1	10	10
30. Tetrachloroethene	127-18-4	5	5
31. Toluene	108-88-3	5	5
32. Chlorobenzene	108-90-7	5	5
33. Ethyl Benzene	100-41-4	5	5
34. Styrene	100-42-5	5	5
35. Total Xylenes		5	5

[a] Medium Water Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Water CRDL.

[b] Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Soil/Sediment CRDL.

Semi-Volatiles	CAS Number	Detection Limits* Low Water[c] ug/L	Low Soil/Sediment[d] ug/Kg
36. Phenol	108-95-2	10	330
37. bis(2-Chloroethyl) ether	111-44-4	10	330
38. 2-Chlorophenol	95-57-8	10	330
39. 1,3-Dichlorobenzene	541-73-1	10	330
40. 1,4-Dichlorobenzene	106-46-7	10	330
41. Benzyl Alcohol	100-51-6	10	330
42. 1,2-Dichlorobenzene	95-50-1	10	330
43. 2-Methylphenol	95-48-7	10	330
44. bis(2-Chloroisopropyl) ether	39638-32-9	10	330
45. 4-Methylphenol	106-44-5	10	330
46. N-Nitroso-Dipropylamine	621-64-7	10	330
47. Hexachloroethane	67-72-1	10	330
48. Nitrobenzene	98-95-3	10	330
49. Isophorone	78-59-1	10	330
50. 2-Nitrophenol	88-75-5	10	330
51. 2,4-Dimethylphenol	105-67-9	10	330
52. Benzoic Acid	65-85-0	50	1600
53. bis(2-Chloroethoxy) methane	111-91-1	10	330
54 2,4-Dichlorophenol	120-83-2	10	330
55. 1,2,4-Trichlorobenzene	120-82-1	10	330
56. Naphthalene	91-20-3	10	330
57. 4-Chloroaniline	106-47-8	10	330
58. Hexachlorobutadiene	87-68-3	10	330
59. 4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	330
60. 2-Methylnaphthalene	91-57-6	10	330
61. Hexachlorocyclopentadiene	77-47-4	10	330
62. 2,4,6-Trichlorophenol	88-06-2	10	330
63. 2,4,5-Trichlorophenol	95-95-4	50	1600

(continued

Semi-Volatiles	CAS Number	Detection Limits* Low Water[c] ug/L	Low Soil/Sediment[d] ug/Kg
64. 2-Chloronaphthalene	91-58-7	10	330
65. 2-Nitroaniline	88-74-4	50	1600
66. Dimethyl Phthalate	131-11-3	10	330
67. Acenaphthylene	208-96-8	10	330
68. 3-Nitroaniline	99-09-2	50	1600
69. Acenaphthene	83-32-9	10	330
70. 2,4-Dinitrophenol	51-28-5	50	1600
71. 4-Nitrophenol	100-02-7	50	1600
72. Dibenzofuran	132-64-9	10	330
73. 2,4-Dinitrotoluene	121-14-2	10	330
74. 2,6-Dinitrotoluene	606-20-2	10	330
75. Diethylphthalate	84-66-2	10	330
76. 4-Chlorophenyl Phenyl ether	7005-72-3	10	330
77. Fluorene	86-73-7	10	330
78. 4-Nitroaniline	100-01-6	50	1600
79. 4,6-Dinitro-2-methylphehol	534-52-1	50	1600
80. N-nitrosodiphenylamine	86-30-6	10	330
81. 4-Bromophenyl Phenyl ether	101-55-3	10	330
82. Hexachlorobenzene	118-74-1	10	330
83. Pentachlorophenol	87-86-5	50	1600
84. Phenanthrene	85-01-8	10	330
85. Anthracene	120-12-7	10	330
86. Di-n-butylphthalate	84-74-2	10	330
87. Fluoranthene	206-44-0	10	330
88. Pyrene	129-00-0	10	330
89. Butyl Benzyl Phthalate	85-68-7	10	330
90. 3,3'-Dichlorobenzidine	91-94-1	20	660
91. Benzo(a)anthracene	56-55-3	10	330
92. bis(2-ethylhexyl)phthalate	117-81-7	10	330
93. Chrysene	218-01-9	10	330
94. Di-n-octyl Phthalate	117-84-0	10	330
95. Benzo(b)fluoranthene	205-99-2	10	330
96. Benzo(k)fluoranthene	207-08-9	10	330
97. Benzo(a)pyrene	50-32-8	10	100

(continued)

Semi-Volatiles	CAS Number	Detection Limits* Low Water[c] ug/L	Low Soil/Sediment[d] ug/Kg
98. Indeno(1,2,3-cd)pyrene	193-39-5	10	330
99. Dibenz(a,h)anthracene	53-70-3	10	330
100. Benzo(g,h,i)perylene	191-24-2	10	330

[c]Medium Water Contract Required Detection Limits (CRDL) for Semi-Volatile HSL Compounds are 100 times the individual Low Water CRDL.

[d]Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Semi-Volatile HSL Compounds are 60 times the individual Low Soil/Sediment CRDL.

Table 1. Elements Determined by Inductively Coupled Plasma Emission or Atomic Absorption Spectroscopy

Element	Contract Required Detection Level[1,2] (ug/L)
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium total (+3, +6)	10
Cobalt	50
Copper	25
Iron	100
Lead	5
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Vanadium	50
Zinc	20
Cyanide	5

Attachment B
SAMPLING AND ANALYSIS

Table B-1

PRELIMINARY DESIGN SAMPLING AND ANALYSIS AT NORTHSIDE SANITARY LANDFILL

TASK	SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	SAMPLE NO.	SAMPLE FREQ.	SAMPLE TOTAL	REPLICATES (4) NO.	REPLICATES (4) FREQ.	REPLICATES (4) TOTAL	FIELD BLANKS (5) NO.	FIELD BLANKS (5) FREQ.	FIELD BLANKS (5) TOTAL	MATRIX SPIKE (6) NO.	MATRIX SPIKE (6) FREQ.	MATRIX SPIKE (6) TOTAL	MATRIX TOTAL
FT-SOURCE TESTING	LEACHATE	pH Specific Conductance Temperature Sulfide spot test	VOC's consistent with RAS Protocol (1) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			A,B/N Extractables consistent with RAS Protocol (1) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Total and Soluble Metals RAS Protocol (1) Filtered and Unfiltered Samples	30	1	30	3	1	3	3	1	3	3	1	3	36
			Cyanide consistent with RAS Protocol (1) or SAS Protocol (3) Unfiltered Samples (see note 7)	15	1	15	2	1	2	2	1	2	2	1	2	19
			Alkalinty see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Solids, Non-Filterable(Suspended) see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Solids, Filterable(Dissolved) see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Solids, Volatile (Suspended) see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Nitrogen, Nitrate + Nitrite see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Nitrogen, Ammonia see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Chloride see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Phosphorous, Total see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Nitrogen, Total Kjeldahl see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Oil and Grease see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Sulfate see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			BOD-5 see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Chemical Oxygen Demand see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19
			Total Organic Carbon see ISBH Protocol (2) Unfiltered Samples	15	1	15	2	1	2	2	1	2	2	1	2	19

Table B-1
(p 2 of 2)

PRELIMINARY DESIGN SAMPLING AND ANALYSIS AT NORTHSIDE SANITARY LANDFILL

TASK	SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	SAMPLE NO.	SAMPLE FREQ.	SAMPLE TOTAL	REPLICATES (4) NO.	REPLICATES (4) FREQ.	REPLICATES (4) TOTAL	FIELD BLANKS (5) NO.	FIELD BLANKS (5) FREQ.	FIELD BLANKS (5) TOTAL	MATRIX SPIKE (6) NO.	MATRIX SPIKE (6) FREQ.	MATRIX SPIKE (6) TOTAL	MATRIX TOTAL
FT-SOURCE TESTING	GROUNDWATER	pH	VOC's consistent with RAS Protocol (1) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
		Specific Conductance	A,B/N Extractables consistent with RAS Protocol (1) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
		Temperature	Total and Soluble Metals RAS Protocol (1) Filtered and Unfiltered Samples	50	1	50	5	1	5	5	1	5	5	1	5	60
		Sulfide spot test	Cyanide consistent with RAS Protocol (1) or SAS Protocol (3) Unfiltered Samples (see note 7)	25	1	25	3	1	3	3	1	3	3	1	3	31
			Alkalinity see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			Solids, Non-Filterable(Suspended) see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			Solids, Filterable(Dissolved) see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			Solids, Volatile (Suspended) see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			Nitrogen, Nitrate + Nitrite see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			Nitrogen, Ammonia see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			Chloride see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			Phosphorous, Total see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			Nitrogen, Total Kjeldahl see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			Oil and Grease see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			Sulfate see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			BOD-5 see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			Chemical Oxygen Demand see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31
			Total Organic Carbon see ISBH Protocol (2) Unfiltered Samples	25	1	25	3	1	3	3	1	3	3	1	3	31

TABLE B-1

(P 3 of 3)

Note:
1. See Attachment of Appendix A for a complete list of parameters.
2. See ISBH Protocol in Appendix D.
3. See CLP/SAS Protocol in Appendix C.
4. Replicate to be collected at 10% or one per day, whichever is greater.
5. Field blanks to be collected at 10% or one per day, whichever is greater.
6. For VOC samples, a trip blank will be shipped with each set of samples.
7. For matrix spike and matrix spike duplicate, the VOC samples will be collected at double of the normal required volumes while the extractable samples will be collected at triple of the normal required volumes.
8. Selection of cyanide determination (by RAS or SAS) depends on the outcome of the sulfide spot test and may require field filtration.

Attachment C
SAMPLE QUANTITIES, BOTTLES, AND PRESERVATIVES

TABLE C-1
SAMPLE TYPES, BOTTLES, AND PRESERVATIVES
NSL/ECC SITE

Sample Type	Analysis	Bottles	Preservation	Holding Time	Quantity	Method of Shipment	Packing
Aqueous Low level	CLP/RAS Organics						
	- Acid extractables Base/neutral	Two 1/2-gallon amber bottles (Teflon lined-caps)	Iced to 4 C	5 days for extraction 40 days for analysis	Fill to Shoulder	Daily by overnight carrier	Vermiculite or Poly-foam cooler
	- Volatiles	Two 40-ml volatile organic analysis vials	Iced to 4 C	7 days	Fill to top, no air space	Daily by overnight carrier	Vermiculite or Poly-foam cooler
	CLP/RAS Inorganics						
	- Metals (including mercury)	Two 1-liter polyethylene bottle	HNO3 to pH (2, Iced to 4 C	6 months (30 days for Mercury)	Fill to Shoulder	Daily by overnight carrier	Vermiculite or Poly-foam cooler
	- Cyanide	One 1-liter polyethylene bottle	NaOH to pH) 12 Iced to 4 C	14 days	Fill to Shoulder	Daily by overnight carrier	Vermiculite or Poly-foam cooler
	CLP/SAS Inorganics						
	- Cyanide	One 1-liter polyethylene bottle	Cadmium Carbonate NaOH to pH) 12 Iced to 4 C	14 days	Fill to Shoulder	Daily by overnight carrier	Vermiculite or Poly-foam cooler
	ISBH/SAS Conventional Parameters						
	- BOD	One 1-liter polyethylene bottle	Iced to 4 C	48 hours	Fill to Shoulder	Daily by overnight carrier	Vermiculite or Poly-foam cooler
	- COD, TOC, nitrate + nitrite Total Kjeldahl Nitrogen, Ammonia, Total phosphorous	One 1-liter polyethylene bottle	H2SO4 to pH (2, Iced to 4 C	28 days	Fill to Shoulder	Daily by overnight carrier	Vermiculite or Poly-foam cooler
	- Total suspended solids Volatile suspended solids Total dissolved solids Alkalinity Chlorides Sulfates	One 1-liter polyethylene bottle	Iced to 4 C	7 days 7 days 48 hours 48 hours 28 days 28 days	Fill to Shoulder	Daily by overnight carrier	Vermiculite or Poly-foam cooler
Aqueous Medium	RAS Organics						
	- Acid extractables Base/neutral	Two 1/2-gallon amber bottles (Teflon lined-caps)	Iced to 4 C	5 days for extraction 40 days for analysis	Fill to Shoulder	Federal Express Priority 1 with restricted article paperwork	In cans with vermiculite
	- Volatiles	Two 40-ml volatile organic analysis vials	Iced to 4 C	7 days	Fill to top, no air space	Federal Express Priority 1 with restricted article paperwork	In cans with vermiculite
	RAS Inorganics						
	- Metals (including mercury)	One 1-liter polyethylene bottle	HNO3 to pH (2, Iced to 4 C	6 months (30 days for Mercury)	Fill to Shoulder	Federal Express Priority 1 with restricted article paperwork	In cans with vermiculite
	SAS						
	- BOD	One 1-liter polyethylene bottle	Iced to 4 C	48 hours	Fill to Shoulder	Federal Express Priority 1 with restricted article paperwork	In cans with vermiculite
	- COD, TOC, nitrate + nitrite	One 1-liter polyethylene bottle	H2SO4 to pH (2, Iced to 4 C	28 days	Fill to Shoulder	Federal Express Priority 1 with restricted article paperwork	In cans with vermiculite
	- Total suspended solids Volatile suspended solids Total dissolved solids Alkalinity Chlorides Sulfates	One 1-liter polyethylene bottle	Iced to 4 C	7 days 7 days 48 hours 48 hours 28 days 28 days	Fill to Shoulder	Federal Express Priority 1 with restricted article paperwork	In cans with Poly-foam cooler vermiculite

Appendix B
EXISTING DATA

TABLE 9

NORTHSIDE SANITARY LANDFILL
LIQUID LEACHATE RESULTS
PHASE III - SAMPLING
REMEDIAL INVESTIGATION
LIQUID LEACHATE

Sample Location:	LEACHATE TANK 1	LEACHATE TANK 1	LEACHATE TANK 2	LEACHATE TANK 3	
Sample Number:	NSL-LL003-02	NSL-LL007-02	NSL-LL006-02	NSL-LL004-02	NSL-LL008-02
Sample Type:		DUP NSL-LL005-02			FIELD BLANK
Date Sampled:	11/21/85	11/21/85	11/21/85	11/21/85	11/21/85
OTR Number:	EE365	EE367	EE366	EE364	EE368
ITR Number:	ME6200	MEM602	MEM601	ME6199	MEM603
ORGANIC COMPOUNDS (ug/l)					
VOLATILES					
BENZENE	5	9 J		7	
CHLORODENZENE				6	
1, 1-DICHLORDETHANE			460		
TRANS-1, 2-DICHLORDETHENE			1300		
ETHYLBENZENE	100	220		84	
METHYLENE CHLORIDE			2200	7	
TOLUENE	21	23		55	
ACETONE			11000 J	120 J	
2-BUTANONE	15		12000	93	
4-METHYL-2-PENTANONE	11		2000	72	
TOTAL XYLENES	540	1100	11000	480	
TOTAL VOLATILES	692	1352	39960	926	
TOTAL TENTATIVELY IDENTIFIED VOLATILES	542	700	706	330	0
BASE NEUTRALS and ACIDS					
4-CHLORO-3-METHYLPHENOL				15 J	
2, 4-DIMETHYLPHENOL		13 J			
PHENOL	7 J	15 J	370 J		
BENZOIC ACID			1460 J	180 J	
4-METHYLPHENOL			1350 J	37 J	
ISOPHORONE				73 J	
NAPHTHALENE	23 J	16 J			
BIS(2-ETHYLHEXYL)PHTHALATE	24 J	64 J	650 J	20 J	
DI-N-BUTYL PHTHALATE				3 J	
DIETHYL PHTHALATE	24 J	21 J	71 J	27 J	
TOTAL BASE NEUTRALS and ACIDS	78 J	129 J	3901 J	355 J	
TOTAL TENTATIVELY IDENTIFIED ACIDS BASE/NEUTRALS	4149	2439	10270	1864	15
PESTICIDES and PCBs	UNUSABLE				
TOTAL PESTICIDES and PCBs	0	0	0	0	

TABLE 9

NORTHSIDE SANITARY LANDFILL
LIQUID LEACHATE RESULTS
PHASE III - SAMPLING
REMEDIAL INVESTIGATION
LIQUID LEACHATE

Sample Location:	LEACHATE TANK 1	LEACHATE TANK 1	LEACHATE TANK 2	LEACHATE TANK 3	
Sample Number:	NSL-LL005-02	NSL-LL007-02	NSL-LL006-02	NSL-LL004-02	NSL-LL008-02
Sample Type:		DUP NSL-LL005-02			FIELD BLANK
Date Sampled:	11/21/85	11/21/85	11/21/85	11/21/85	11/21/85
QTR Number:	EE345	EE347	EE346	EE344	EE348
ITR Number:	MEN200	MEN602	MEN601	MEN199	MEN603
INORGANIC COMPOUNDS (ug/l)					
ALUMINUM	[133]	[183]	354	[74]	
ARSENIC			11		
BARIUM	782	732	[117]	349	
CALCIUM	152000	159000	262000	219000	
CHROMIUM	15	16	14	10	
COBALT	[11]	[9.7]	[11]	[12]	
COPPER	[14]	[14]	[21]	28	[7.8]
IRON	21400	24300	36800	44100	[41]
LEAD	39	31	28	22	8.7
MAGNESIUM	175000	176000	135000	88900	
MANGANESE	185	223	827	731	
NICKEL	99	101	58	[39]	
POTASSIUM	332000	331000	212000	145000	
SILVER			[5.1]	[3.8]	
SODIUM	629000	623000	385000	206000	
VANADIUM	[4.7]	[5.2]	[7.1]		
ZINC	271	108	149	157	[7.4]
	(mg/l)				
OIL AND GREASE	<5	<5	37	<5	<5

FOOTNOTES:
- J: Estimated value.
- B: Used when the analyte is found in the laboratory sample. Indicates possible/probable contamination.
- Z: Coelution with other compounds prevents spectral confirmation according to contact guidelines; this compound is believed to be present.
- [ ]: Positive values less than the contract required detection limit.

TABLE A-7
NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
GLACIAL TILL WATER BEARING UNIT
PHASE I SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Location:	NSL08A	NSL09S	NSL10S	NSL11S	NSL14	NSL15	NSL16	NSL18			
Sample Number:	GW08S-01	GW09S-01	GW10S-01	GW11S-01	GW014-01	GW015-01	GW016-01	GW018-01	GW022-01	GW023-01	GW024-01
Sample Type:									FIELD BLANK	FIELD BLANK	FIELD BLANK
Date Sampled:	4-15-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-21-85	4-15-85
QTR Number:	ED167	EA344	EA346	EA348	EA352	EA353	EA354	EA355	EA361		ED169
ITR Number:	MED357	MEC532	MEC534	MEC536	MEC540	MEC541	MEC542	MEC543	MEC549	MEC550	MED359
ORGANIC COMPOUNDS (ug/l)											
VOLATILES											
2-BUTANONE		10 J					1000 J			TESTED FOR	
ACETONE	11	110	750 B	11000 B	100 J	810	13000	14	10 J,B	INORGANICS	16
BENZENE							500 J	5 J		ONLY	
CHLORDETHANE		10 J									
ETHYLBENZENE					50 J			5 J			
METHYLENE CHLORIDE		5 J	50 J		50 J,B	50 J,B	500 J,B	5 J	5 J		
TOLUENE		5 J	50 J	500 J	50 J		500 J	19			
TOTAL XYLENES	2.6 J	5 J	50 J		290	50 J	830	62			
TRICHLORDETHENE		5 J									
4-METHYL-2-PENTANONE	1.1 J										
TOTAL VOLATILES	14.7	145	900	11500	540	910	16330	110	15		16
TOTAL TENTATIVELY IDENTIFIED VOLATILES	0	59.6 J	180 J	800 J	61 J	0	0	0	0		15 J
BASE/NEUTRALS and ACIDS											
2-METHYLNAPHTHALENE	8.1 J		10 J			65	110	10 J			
2-METHYLPHENOL			10 J								
4-METHYLPHENOL			10 J								
BIS(2-ETHYLHEXYL)PHTHALATE				12 J	10 J	10 J	14	10 J			
DI-N-BUTYL PHTHALATE			10 J,B	12 J,B	10 J,B	10 J,B	10 J,B	10 J,B	10 J,B		
NAPHTHALENE			10 J			33	28	10 J			
PHENOL			10 J								
PHENANTHRENE							10 J				
TOTAL BASE/NEUTRALS and ACIDS	8.1	0	60	20	20	118	172	40	10		0
TOTAL TENTATIVELY IDENTIFIED BASE/NEUTRALS and ACIDS	276 J	70.3 J	157.8 J	972 J	5195 J	709 J	2863 J	21 J			26 J
PESTICIDES and PCBs											
TOTAL PESTICIDES and PCBs	0	0	0	0	0	0	0	0	0		0

TABLE A-7
NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
GLACIAL TILL WATER BEARING UNIT
PHASE I SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Location:	NSL05R	NSL09	NSL10S	NSL11S	NSL14	NSL15	NSL16	NSL18			
Sample Number:	GW05S-01	GW09S-01	GW10S-01	GW11S-01	GW014-01	GW015-01	GW016-01	GW018-01	GW022-01	GW023-01	GW024-01
Sample Type:									FIELD BLANK	FIELD BLANK	FIELD BLANK
Date Sampled:	4-15-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-21-85	4-15-85
OTR Number:	ED167	EA344	EA346	EA348	EA352	EA353	EA354	EA355	EA361		ED169
ITR Number:	MED357	MEC532	MEC534	MEC536	MEC540	MEC541	MEC542	MEC543	MEC549	MEC550	MED359
INORGANIC COMPOUNDS (ug/l)											
ALUMINUM	[63]			[24]							
ANTIMONY					[32] R			[33] R			
ARSENIC					21 +,S						
BARIUM	[169]	362	[111]	[84]	1110	[69]	[94]	[176]		[1.2]	
BERYLLIUM								[1.2]	[1.3]		
CALCIUM	54000	167000	228000	86600	182000	77300	91300	181000	[92]	[24]	
CHROMIUM		[3.7]									
COBALT		[9.4]	[7.7]	[6.1]	[9.8]			[17]			
COPPER		[8.3]							[9.4]		
IRON	[79]	[74]	[18]	[60]	17000	[23]	[8.2]	797	[30]	[6.6]	
LEAD	9.4 R			30		26	53				
CYANIDE		[2.5]	NR					[3.9]			
MAGNESIUM	27700	98000	67400	26600	165000	22900	38500	73700	[36]	[6.5]	
MANGANESE	148 *	498	912	112	654	204	185	4330			[3.2] *
NICKEL		87	[32]		55			[35]			
POTASSIUM	5400	13500	10600	[4960]	152000	[1450]	[2620]				
SILVER									[4.9] R		
SODIUM	26600	335000	142000	30400	534000	6930	14200	38000	[60]	[125]	[55.2]
VANADIUM					[3.6]			[4.8]			
ZINC	[19]	[8.8]	290	[16]	[3.7]	[17]		[16]	[5.6]		[7.7]
pH	7.6	6.7	NR	7.6	6.7	7.6	7.2	6.5	6	7	6.4
PERCENT SOLIDS (%)	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
OIL AND GREASE (mg/l)	<8	16.3		3.5	18.2	6.46	49.5	11.5			
TOTAL ALKALINITY (mg/l as CaCO3)	5750	1406		967	1933.7	607	981.5	882.4			
CHLORIDE (mg/l)	5	625		25	966	23.6	21.6	100			
DISSOLVED SOLIDS (mg/l)	390	1751		445	2935	202	339	823		63	20
SULFATE (mg/l)		54.5		96.7	10.5	19.1	19.1	172			

FOOTNOTES:

B: Analyte has been found in the laboratory blank as well as the sample. Indicates possible/probable contamination.
C: Applies to pesticide parameters where the identification has been confirmed by GC/MS.
E: Value is estimated due to presence of interference.
J: An estimated value.
K: Actual value, within the limitations of the method, is less than the given value.
R: Spike sample recovery is not within control limits.
S: Value is determined by standard addition.
*: Duplicate analysis is not within control limits.
+: Sample(s) analyzed at medium concentrations.
+: Correlation coefficient for method of standard addition is less than 0.995.
[]: Positive values less than the contract required detection limit.
NR: Not required by contract at this time.

10-Feb-86

TABLE A-7
NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
GLACIAL TILL WATER BEARING UNIT
PHASE II SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Location:	NSL8SA	NSL9S	NSL10S	NSL11S	NSL14	NSL15	NSL16	NSL18		
Sample Number:	GW0086-02	GW0095-02	GW0105-02	GW0115-02	GW0114-02	GW0115-02	GW0116-02	GW0118-02	GW0127-02 FIELD BLANK	GW0118-02 FIELD DUPLICATE
Sample Type:										
Date Sampled:	5-14-85	5-15-85	5-14-85	5-14-85	5-15-85	5-15-85	5-14-85	5-15-85	5-14-85	5-15-85
QTR Number:	EDC65	EDC17	EDC19	EDC21	EDC25	EDC26	EDC27	EDC28	EDC72	EDC34
ITR Number:	MED137	MED139	MED141	MED143	MED147	MED148	MED149	MED150	MED585	MED586
ORGANIC COMPOUNDS (ug/l)										
VOLATILES										
CARBON DISULFIDE							1.1 J			
BENZENE							22			
TOTAL XYLENES				5.1	44		1100			
TRICHLOROETHENE			5 J	5 J		32				
ACETONE		10 BJ	10 JB	10 JB	510 BJ		550 BJ			
2-BUTANONE		10 BJ			370 BJ		12 BJ			
METHYLENE CHLORIDE	7.2		6.6 B	15 B	13 B				7.5	
4-METHYL-2-PENTANONE					110 J					
TOLUENE							28			
TOTAL VOLATILES	7.2	20	21.6	35.1	1047	32	1713.1	0	7.5	0
TOTAL TENTATIVELY IDENTIFIED VOLATILES	0	0	0	0	250.1 J	0	0	0	0	0
TOTAL PESTICIDES and PCBs	0	0	0	0	0	0	0	0	0	0
BASE/NEUTRALS and ACIDS										
PHENOL				10 J			3.6 J			3.9 J
2-METHYLPHENOL							9.9 J			
2-METHYLNAPHTHALENE				31	110		98			
DI-N-BUTYL PHTHALATE				3 J						
BIS(2-ETHYLHEXYL)PHTHALATE		26		10 J	13	10 JB				
NAPHTHALENE				24	99		110			
TOTAL BASE/NEUTRALS AND ACIDS	0	0	26	85	222	10	213.5	0	0	3.9
TOTAL TENTATIVELY IDENTIFIED BASE/NEUTRALS and ACIDS	75 J	182 J	0	1646 J	4953.5 J	465 J	5417.5 J	186.8 J	28 J	17.5 J

10-Feb-86

TABLE A-7
NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
GLACIAL TILL WATER BEARING UNIT
PHASE II SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Location	[redacted]	NSL9S	NSL10S	NSL11S	NSL14	NSL15	NSL16	NSL18		
Sample Number:	GW008S-02	GW009S-02	GW010S-02	GW011S-02	GW014-02	GW015-02	GW016-02	GW018-02	GW027-02 FIELD BLANK	GW028-02 FIELD BLANK
Sample Type:										
Date Sampled:	5-14-85	5-15-85	5-14-85	5-14-85	5-15-85	5-15-85	5-14-85	5-15-85	5-14-85	5-15-85
QTR Number:	ED669	ED617	ED619	ED621	ED625	ED626	ED627	ED628	ED672	ED634
ITR Number:	MED137	MED139	MED141	MED143	MED147	MED148	MED149	MED150	MED505	MED506
INORGANIC COMPOUNDS (ug/l)										
ALUMINUM			306							
ANTIMONY										70
ARSENIC					44 R					
BARIUM		400			900					
CALCIUM	51000	210000	207000	70000	264000	81000	68000	184000		
IRON					22200			2230		
LEAD				50		14	20			
MAGNESIUM	25000	106000	59000	21000	152000	22000	23000	65000		
MANGANESE	50	580	1030	240	680	250	50	4330		
NICKEL		100			70					
POTASSIUM		11000	6000	[3000]	156000					
SODIUM	25000	360000	75000	28000	495000	9000	13000	41000		
PH (units)	7.7	7.2	7.2	7.6	6.9	7.6	7.8	6.8	8.85	8.85
PERCENT SOLIDS	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
OIL AND GREASE (mg/l)	2	2	4	3	33	3	4	1		
TOTAL ALKALINITY(mg/l as CaCO3)	525	1640	876	1942	1808	624	572	692	2	4
CHLORIDE (mg/l)	10	694	35	15	889	16	26	71		
DISSOLVED SOLIDS (mg/l)	420	2240	1330	684	3010	524	466	1000	12	34
SULFATE (mg/l)		16	477	77	25	54	77	157		

FOOTNOTES:
S: Value determined by method of standard addition.
R: Spike sample recovery is not within control limits.
J: An Estimated Value.
[]: Value is greater than or equal to the instrument detection limit but less than the contract required detection limit.
NR: Not required by contract at this time.

TABLE 3.8
NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
SAND AND GRAVEL WATER BEARING UNIT
PHASE I SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Locations:	MW1	MW1	MW2	MW3	MW4	MW5	MW6	MW8	MW7	MW9
Sample Number:	GW001 01	GW017 01 GW001-01 DUP	GW002 01	GW003 01	GW004 01	GW005 01	GW006 01	GW008 01 GW006-01-DUP	GW007 01	GW009 01 GW007-01 DUP
Sample Type:										
Date Sampled:	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85
QUA Number:	EA562	EA558	EA563	EA564	EA565	EA339	EA340	EA395	EA341	EA396
ITR Number:	ME4864	ME3502	ME3503	ME3505	ME3506	ME3527	ME3528	ME3547	ME3529	ME3548
ORGANIC COMPOUNDS (ug/l)										
VOLATILES										
1, 1, 1-TRICHLOROETHANE										
1, 1-DICHLOROETHANE		8.7				5 J				
1, 1-DICHLOROETHENE										
ACETONE	100 J	17 B	10 J,B	10 J	10 J	10 J	10 J			10 J
BENZENE		5 J								
CHLOROETHANE	100 J	10 J								
ETHYLBENZENE										
METHYLENE CHLORIDE	50 J,B	5 J	5 J	5 J,B		5 J	5 J,B	5 J,B		5 J
TOLUENE										
TOTAL XYLENES										
TRANS-1,2-DICHLOROETHENE		5 J				5 J				
TRICHLOROETHENE		5 J								
VINYL CHLORIDE	120 J,B	10 J								
4-METHYL-2-PENTANONE										
TOTAL VOLATILES	350	65.7	15	15	10	25	15	5	0	15
TOTAL TENTATIVELY IDENTIFIED VOLATILES	0	74.6 J	12 J	0	0	0	10 J	0	0	0
BASE/NEUTRALS and ACIDS										
2-METHYLNAPHTHALENE										
2-METHYLPHENOL										
4-METHYLPHENOL										
BIS(2-ETHYLHEXYL)PHTHALATE										
BUTYL BENZYL PHTHALATE					10 J					
DI-N-BUTYL PHTHALATE	10 J,B	10 J,B	5 J,B	10 J,B	10 J,B	10 J,B	10 J,B	10 J,B	10 J,B	10 J,B
DIETHYL PHTHALATE			10 J							
NAPHTHALENE										
PHENOL										
TOTAL BASE/NEUTRALS and ACIDS	10	10	20	10	20	10	10	10	10	10
TOTAL TENTATIVELY IDENTIFIED BASE/NEUTRALS and ACIDS	216 J	238.2 J	12 J	21 J	0	0	16.9 J	53 J	25 J	0
PESTICIDES and PCBs										
ALDRIN										
TOTAL PESTICIDES and PCBs	0	0	0	0	0	0	0	0	0	0

10-Feb-86

TABLE A-8
NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
SAND AND GRAVEL WATER BEARING UNIT
PHASE I SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Location:	MW1	MW1	MW2	MW3	MW4	MW5	MW6	MW6	MW7	MW7
Sample Number:	GW001-01	GW019-01	GW002-01	GW003-01	GW004-01	GW005-01	GW006-01	GW20E-01	GW007-01	GW007-01-DUP
Sample Type:		GW001-01-DUP						GW20E-01-DUP		
Date Sampled:	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85
OTA Number:	E9262	EA358	E9263	E9264	E9265	EA339	EA340	EA359	EA341	EA342
ITA Number:	ME486A	MEC546	ME486S	MEC525	MEC526	MEC527	MEC528	MEC547	MEC529	MEC548
INORGANIC COMPOUNDS (ug/l)										
ALUMINUM						[114]				
ANTIMONY		[35] R								
ARSENIC	15 *,S	15								
BARIUM	1490	1460	243	211	[152.]	[171]	311	299	229	240
BERYLLIUM										
CALCIUM	89300	86000	53600	43700	25000	58600	69100	64400	75000	60400
CHROMIUM		[4.4]				[3.9]	[3.0]			
COBALT	[8.7]	[11]								
COPPER					[2.9]	[3.0]	[2.6]		[2.6]	
IRON	7530	6730	363	[11]	[38]	[71]	486	446	262	276
LEAD										
CYANIDE										
MAGNESIUM	114000	110000	20600	23500	12400	26000	30700	26900	26500	25200
MANGANESE	150	147	58	81	[12]	36	37	37	72	67
NICKEL	87	80								
POTASSIUM	106000	107000		[1460]	[1240]	[717]	[1670]	[592]	[668]	
SILVER			[3.1] R					[3.8] R	[4.0] R	
SODIUM	412000	410000	37200	35000	62200	6540	21700	20800	14300	14000
TIN									[15] R	
VANADIUM										
ZINC	41	42	[17]	[5.7]	[5.9]		[3.4]	[3.2]	[11]	
pH	6.9	7	7.6	7.7	7.6	7.3	7.2	7.3	7.1	7.1
PERCENT SOLIDS (%)	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
OIL AND GREASE (mg/l)	0.8]	1.0]		26.6	26	26.7	24	0.45	3.7	0.43
TOTAL ALKALINITY (mg/l as CaCO3)	917.6	958.7	259.5	274.4	303.5	305.5	361.9	371.9	335.7	344.7
CHLORIDE (mg/l)	761	821	5.7	6.3	7	14	1.7	1.7	10.6	12.7
DISSOLVED SOLIDS (mg/l)	2137	2104	239	376	366	333	246	204	283	305
SULFATE (mg/l)	15	13.8	1		1	96.7	1	1.4	20.7	22.1

FOOTNOTES:

B: Analyte has been found in the laboratory blank as well as the sample. Indicates possible/probable contamination.
J: An estimated value.
R: Spike sample recovery is not within control limits.
S: Value is determined by standard addition.
*: Duplicate analysis is not within control limits.
@: Analyzed at Conc./Dil Factor different from other volatiles.
+: Correlation coefficient for method of standard addition is less than 0.995.
[]: Positive values less than the contract required detection limit.
NR: Not required by contract at this time.

TABLE 4.8
NORTHSIDE SANITARY LANDFILL MONITORING WELL NETWORK
SAND AND GRAVEL WATER BEARING UNIT
PHASE I SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Location:	NSL8DA	NSL9D	NSL10D	NSL11D	NSL12	NSL13	SB#65			
Sample Number:	GW08D-01	GW09D-01	GW10D-01	GW11D-01	GW012-01	GW013-01	GW065-01	GW023-01 FIELD BLANK	GW023-01 FIELD BLANK	GW024-01 FIELD BLANK
Sample Type:										
Date Sampled:	4-15-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-21-85	4-15-85
QTR Number:	ED168	EA345	EA347	EA349	EA350	EA351	EA356	EA361		ED169
ITR Number:	MED358	MEC533	MEC535	MEC537	MEC538	MEC539	MEC544	MEC549	MEC550	MED359
ORGANIC COMPOUNDS (ug/l)										
VOLATILES										
1, 1, 1-TRICHLORDETHANE					7.3	5 J			TESTED FOR INORGANICS ONLY	
1, 1-DICHLORDETHANE						60				
1, 1-DICHLORDETHENE						5 J				
2-BUTANONE			20 J		13					
ACETONE	4.6 J	310	710	41 B	19	10 J	40 B	10 J,B		16
BENZENE					5 J	5 J	110			
CHLORDETHANE					33	20	10 J			
ETHYLBENZENE						5 J	11			
METHYLENE CHLORIDE		5 J		5 J	5 J,B	5 J,B	5 J	5 J		
TOLUENE							5 J			
TOTAL XYLENES	1.1 J	5 J			6.6		9.4			
TRANS-1,2-DICHLORDETHENE					11	70				
TRICHLORDETHENE			10 J		5.2	8				
VINYL CHLORIDE					10 J	32				
4-METHYL-2-PENTANONE										
TOTAL VOLATILES	5.7	320	740	46	115.1	225	190.4	15		16
TOTAL TENTATIVELY IDENTIFIED VOLATILES	0	0	0	0	113.1 J	27 J	134 J	0		15 J
BASE/NEUTRALS and ACIDS										
2-METHYLNAPHTHALENE	3.8 J		10 J							
2-METHYLPHENOL										
4-METHYLPHENOL										
BIS(2-ETHYLHEXYL)PHTHALATE		10 J				10 J				
BUTYL BENZYL PHTHALATE										
DI-N-BUTYL PHTHALATE		10 J,B		10 J,B			10 J,B	10 J,B		
DIETHYL PHTHALATE						10 J				
NAPHTHALENE			10 J							
PHENOL										
TOTAL BASE/NEUTRALS and ACIDS	3.8	20	20	10	0	20	10	10		0
TOTAL TENTATIVELY IDENTIFIED BASE/NEUTRALS and ACIDS	36 J	0	0	0	639.7 J	85 J	493.2 J	0		36 J
PESTICIDES and PCBs										
ALDRIN						0.05 J				
TOTAL PESTICIDES and PCBs	0	0	0	0	0	0.05	0	0		0

TABLE A-8
NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
SAND AND GRAVEL WATER BEARING UNIT
PHASE I SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Location:	NSL8DA	NSL9D	NSL10D	NSL11D	NSL12	NSL13	SB#65			
Sample Number:	GW08D-01	GW09D-01	GW10D-01	GW11D-01	GW12-01	GW13-01	GW065-01	GW22-0. FIELD BLANK	GW23-01 FIELD BLANK	GW24-0. FIELD BLANK
Sample Type:										
Date Sampled:	4-15-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-20-85	2-21-85	4-15-85
QTR Number:	ED168	EA345	EA347	EA349	EA350	EA351	EA356	EA.6:		ED169
ITR Number:	MED358	MEC533	MEC535	MEC537	MEC538	MEC539	MEC544	MEC549	MEC550	MED359
INORGANIC COMPOUNDS (ug/l)										
ALUMINUM	[50]	[77]					[90]			
ANTIMONY		[30] R					[58] R			
ARSENIC	37	12 S					28 S			
BARIUM	[96]	440	[157]	268	830	576	1500		[1.2]	
BERYLLIUM							[1.1]	[1.3]		
CALCIUM	35400	106000	31700	46000	92000	151000	192000	[92]	[24]	
CHROMIUM		[4.9]					[9.9]			
COBALT					[6.7]		[9.0]			
COPPER	[5]							[9.4]		
IRON	[71]	[77]	[39]	[37]	164	1670	10400	[30]	[6.6]	
LEAD	10 R									
CYANIDE		[3.6]					[5.3]			
MAGNESIUM	19500	41800	14900	23200	157000	89500	169000	[36]	[6.5]	
MANGANESE	93 *	125	61	36	348	1380	85			[3.2] *
NICKEL		[24]			84	58	47			
POTASSIUM	[1400]	7400	[3420]	[1750]	55500	27700	147000			
SILVER							[5.6] R	[4.9] R		
SODIUM	32800	119000	69300	35000	426000	250000	536000	[60]	[125]	[1550]
TIN				[13] R						
VANADIUM		[3.3]					[10]			
ZINC	[13]			25		37	21	[5.6]		
PH	7.9	7.1	7.7	7.4	7.2	6.9	6.8	6	7	8.4
PERCENT SOLIDS (%)	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
OIL AND GREASE (mg/l)	20	4.7	5.7	4.35	8.52	1.23	8.75			
TOTAL ALKALINITY (mg/l as CaCO3)	49	2008	348.7	357.6	1050.2	762.8	1155.8			
CHLORIDE (mg/l)	7	243	9	2.3	1030	505	1070			
DISSOLVED SOLIDS (mg/l)	320	749	247	227	2090	1412	3331		63	20
SULFATE (mg/l)		11.6	4.5	1	.4	88.8	6.5			

FOOTNOTES:

B: Analyte has been found in the laboratory blank as well as the sample. Indicates possible/probable contamination.
J: An estimated value.
R: Spike sample recovery is not within control limits.
S: Value is determined by standard addition.
*: Duplicate analysis is not within control limits.
@: Analyzed at Conc./Dil Factor different from other volatiles.
+: Correlation coefficient for method of standard addition is less than 0.995.
[]: Positive values less than the contract required detection limit.
NR: Not required by contract at this time.

10-Feb-86

TABLE 4-8
NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
SAND AND GRAVEL WATER BEARING UNIT
PHASE II SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Location:	MW1	MW2	MW3	MW4	MW5	MW6	MW7	NSL80A	NSL80A
Sample Number:	GW001-02	GW002-02	GW003-02	GW004-02	GW005-02	GW006-02	GW007-02	GW008D-02	GW025-02
Sample Type:									GW008D-02-DUP
Date Sampled:	5-15-85	5-15-85	5-14-85	5-15-85	5-15-85	5-14-85	5-15-85	5-14-85	5-14-85
QTR Number:	ED673	EA370	ED666	EB721	EB722	ED613	ED614	ED670	ED671
ITR Number:	MEC556	MEC557	MEC558	MEC559	MED134	MED135	MED136	MED138	MED503
ORGANIC COMPOUNDS (ug/l)									
VOLATILES									
BENZENE	1.2 J								
CHLORDETHANE	24								
TRICHLORDETHENE	4.4 J								
VINYL CHLORIDE	6.7 J								
ACETONE		9500 BJ		10 JB			24 BJ		
2-BUTANONE		4800 BJ					18 BJ		
METHYLENE CHLORIDE		6.9	6.1	5 JB	5 JB				6.6
TRANS-1, 2-DICHLORDETHENE	23 J								
1, 1, 1-TRICHLORDETHANE	2 J								
1, 1-DICHLORDETHANE	20 J								
4-METHYL-2-PENTANONE		220 J							
ETHYLBENZENE									
TOTAL VOLATILES	81.3	14526.9	6.1	15	5	0	42	0	6.6
TOTAL TENTATIVELY IDENTIFIED VOLATILES	35 J	0	0	0	0	0	0	0	0
TOTAL PESTICIDES and PCBs	0	0	0	0	0	0	0	0	0
BASE/NEUTRALS and ACIDS									
BENZOIC ACID									
DIETHYLPHTHALATE									
DI-N-OCTYL PHTHALATE							1.1 J		
N-NITROSO-DI-N-PROPYLAMINE					1.2 J				
PHENOL									
DI-N-BUTYL PHTHALATE		10 JB							
BIS(2-ETHYLHEXYL)PHTHALATE		10 JB							
TOTAL BASE/NEUTRALS AND ACIDS	0	20	0	0	1.2	0	1.1	0	0
TOTAL TENTATIVELY IDENTIFIED BASE/NEUTRALS and ACIDS	90.9 J	0	42 J	0	0	0	0	22 J	64 J

TABLE A-8
NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
SAND AND GRAVEL WATER BEARING UNIT
PHASE II SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Location:	MW1	MW2	MW3	MW4	MW5	MW6	MW7	NSLBDA	NSLBDA
Sample Number:	GW001-02	GW002-02	GW003-02	GW004-02	GW005-02	GW006-02	GW007-02	GW008D-02	GW035-02 GW028D-02-DUP
Sample Type:									
Date Sampled:	5-15-85	5-15-85	5-14-85	5-15-85	5-15-85	5-14-85	5-15-85	5-14-85	5-14-85
QTR Number:	ED673	EA370	ED666	EB721	EB722	ED613	ED614	ED670	ED671
ITR Number:	MEC556	MEC557	MEC558	MEC559	MED134	MED135	MED136	MED138	MED583
INORGANIC COMPOUNDS (ug/l)									
ANTIMONY									
ARSENIC								28 R	26 R
BARIUM	1400								
CALCIUM	111000	53000	42000	23000	36000	64000	73000	36000	38000
IRON	4970	350				200			
LEAD				6					
CYANIDE									
MAGNESIUM	122000	17000	21000	7000	13000	29000	27000	18000	19000
MANGANESE	160		60					40	50
NICKEL	120								
POTASSIUM	64000								
SODIUM	440000	37000	38000	72000	44000	22000	18000	36000	34000
ZINC									
ph (units)	7	7.4	7.9	7.5	7.6	7.6	7.5	7.8	7.8
PERCENT SOLIDS	NR	NR	NR	NR	NR	NR	NR	NR	NR
OIL AND GREASE (mg/l)									
TOTAL ALKALINITY(mg/l as CaCO3)	1050	330	355	383	264	356	320	307	316
CHLORIDE (mg/l)	871	7	9	5	4		13	7	9
DISSOLVED SOLIDS (mg/l)	2400	360	346	366	300	356	394	338	406
SULFATE (mg/l)	18	13		16			11		11

FOOTNOTES:
S: Value determined by method of standard addition.
R: Spike sample recovery is not within control limits.
J: An Estimated Value.
[]: Value is greater than or equal to the instrument detection limit but less than the contract required detection limit.
NR: Not required by contract at this time.

TABLE A-8
NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
SAND AND GRAVEL WATER BEARING UNIT
PHASE II SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Location:	NSL9D	NSL9D	NSL10D	NSL11D	NSL12	NSL13	SB965	SB977		
Sample Number:	GW0090-02	GW026-02 GW0090-02-DUP	GW010D-02	GW011D-02	GW012-02	GW013-02	GW065-02	GW077-02	GW027-02 FIELD BLANK	GW028-02 FIELD BLANK
Sample Type:										
Date Sampled:	5-15-85	5-15-85	5-14-85	5-14-85	5-14-85	5-14-85	5-15-85	5-14-85	5-14-85	5-15-85
QTR Number:	ED618	ED632	ED620	ED622	ED623	ED624	ED629	ED630	ED672	ED634
ITR Number:	MED140	MED504	MED142	MED144	MED145	MED146	MED501	MED502	MED505	MED506
ORGANIC COMPOUNDS (ug/l)										
VOLATILES										
BENZENE					5 J	1.2 J	120			
CHLOROETHANE					49 J	42 J				
TRICHLOROETHENE			5 J	5 J	5.6	9.2				
VINYL CHLORIDE					4.7 J	72 J				
ACETONE			10 JB	10 JB			110 BJ	10 JB		
2-BUTANONE		11 BJ					92 BJ			
METHYLENE CHLORIDE			5 JB	7.1 B	2.1 J	4.5 J	6 B	5 JB	7.5	
TRANS-1, 2-DICHLOROETHENE					20	130				
1, 1, 1-TRICHLOROETHANE						5.7				
1, 1-DICHLOROETHANE					12	85				
4-METHYL-2-PENTANONE										
ETHYLBENZENE						1.2 J				
TOTAL VOLATILES	0	11	20	22.1	98.4	350.8	328	15	7.5	0
TOTAL TENTATIVELY IDENTIFIED VOLATILES	0	35.9 J	0	0	0	0	222 J	5.7 J	0	0
TOTAL PESTICIDES and PCBs	0	0	0	0	0	0	0	0	0	0
BASE/NEUTRALS and ACIDS										
BENZOIC ACID					7.3 J					
DIETHYLPHTHALATE							2.8 J			
DI-N-OCTYL PHTHALATE										
N-NITROSO-DI-N-PROPYLAMINE										
PHENOL			10 J							3.9 J
DI-N-BUTYL PHTHALATE				10 J				10 J		
BIS(2-ETHYLHEXYL)PHTHALATE										
TOTAL BASE/NEUTRALS AND ACIDS	0	0	10	10	7.3	0	2.8	10	0	3.9
TOTAL TENTATIVELY IDENTIFIED BASE/NEUTRALS and ACIDS	22.4 J	9.5 J	334 J	8 J	881.3 J	284.4 J	2616.3 J	27 J	28 J	17.5 J

TABLE A-8
NORTHSIDE SANITARY LANDFILL MONITORING WELL RESULTS
SAND AND GRAVEL WATER BEARING UNIT
PHASE II SAMPLING
REMEDIAL INVESTIGATION REPORT

Sample Location:	NSL9D	NSL9D	NSL10D	NSL11D	NSL12	NSL13	BB065	SB277		
Sample Number:	GW009D-02	GW026-02	GW010D-02	GW011D-02	GW012-02	GW013-02	GW065-02	GW077-02	GW027-02	GW028-02
Sample Type:		GW009D-02-DUP							FIELD BLANK	FIELD BLANK
Date Sampled:	5-15-85	5-15-85	5-14-85	5-14-85	5-14-85	5-14-85	5-15-85	5-14-85	5-14-85	5-15-85
OTR Number:	ED618	ED632	ED620	ED622	ED623	ED624	ED629	ED634	ED672	ED634
ITR Number:	MED140	MED504	MED142	MED144	MED145	MED146	MED501	MED502	MED505	MED506
INORGANIC COMPOUNDS (ug/l)										
ANTIMONY										70
ARSENIC										
BARIUM					674	570	1600			
CALCIUM	95000	97000	27000	41000	107000	150000	207000	5[illegible]000		
IRON					200	1900	5020			
LEAD				16						
CYANIDE					60					
MAGNESIUM	40000	40000	11000	19000	146000	89000	168000	25000		
MANGANESE	40	40			370	1030		60		
NICKEL					110	70				
POTASSIUM	5000	5000			55000	28000	210000			
SODIUM	120000	103000	72000	34000	384000	264000	575000	2[illegible]000		
ZINC							30 RJ			
pH (units)	7.4	7.4	8	7.7	7.[illegible]	7	6.9	7.6	8.85	8.85
PERCENT SOLIDS	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
OIL AND GREASE (mg/l)										
TOTAL ALKALINITY(mg/l as CaCO3)	744	672	398	422	1500	658	2872	[illegible].6	2	4
CHLORIDE (mg/l)	141	136	11	6	910	634	1050	3		
DISSOLVED SOLIDS (mg/l)	862	892	416	432	2660	2010	3060	294	12	34
SULFATE (mg/l)	16	20			15	61	18			

FOOTNOTES:
S: Value determined by method of standard addition.
R: Spike sample recovery is not within control limits.
J: An Estimated Value.
[]: Value is greater than or equal to the instrument detection limit but less than the contract required detection limit.
NR: Not required by contract at this time.

10-Feb-86

TABLE A-9
PESTICIDE SANITARY LANDFILL RESIDENTIAL WELL RESULTS
RESIDENTIAL WELL SAMPLES
PHASE II SAMPLING

Sample Location:	RW1	RW2	RW3	RW4	RW6	RW5	RW7
Sample Number:	RW001-01	RW002-01	RW003-01	RW004-01	RW006-01	RW005-01	RW007-01
Sample Type:					RW004-01-DUP		FIELD BLANK
Date Sampled:	5-15-85	5-15-85	5-15-85	5-15-85	5-15-85	5-15-85	5-15-85
QTR Number:	ED635	ED636	ED637	ED638	ED640	ED639	ED641
ITR Number:	MED507	MED508	MED509	MED510	MED512	MED511	MED513
ORGANIC COMPOUNDS (ug/l)							
VOLATILES							
METHYLENE CHLORIDE			7.5 B				
2-BUTANONE			16 BJ				
TOTAL VOLATILES	0	2	23.5	0	0	0	0
TOTAL TENTATIVELY IDENTIFIED VOLATILES	0	0	0	0	0	0	0
ACIDS							
PHENOL	2.1 J	6.1 J					
TOTAL ACIDS	2.1	6.1	0	0	0	0	0
TOTAL BASE/NEUTRALS	0	0	0	0	0	0	0
TOTAL TENTATIVELY IDENTIFIED BASE/NEUTRALS and ACIDS	0	0	0	0	0	0	0
TOTAL PESTICIDES and PCBs	0	2	0	0	0	0	0
INORGANIC COMPOUNDS (ug/l)							
CALCIUM	56000	85400		76200	78000	49000	
IRON	600	3300		2750	2600	330	
MAGNESIUM	21000	26000		25000	28000	17000	
MANGANESE						70	
SODIUM	33000	19200	17400	17000	16000	34000	
ZINC						120 RJ	
pH (units)	7.5	7.3	7.8	7.3	7.3	7.5	NR
PERCENT SOLIDS	NR	NR	NR	NR	NR	NR	NR
TOTAL ALKALINITY(mg/l as CaCO3)	302	346	460	360	364	295	4
CHLORIDE (mg/l)	3	9	4	6	7	5	
DISSOLVED SOLIDS (mg/l)	348	428	542	418	414	338	20
SULFATE (mg/l)	16	5	16	35	33	18	13

FOOTNOTES:

B: Analyte has been found in the laboratory blank as well as the sample. Indicates possible/probable contamination.
J: An Estimated Value
R: Spike sample recovery is not within control limits.
NR: Not required by contract at this time.

Appendix C
SPECIAL ANALYTICAL SERVICES

U.S. Environmental Protection Agency
HWI Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[x] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V, NSL/ECC

B. Regional Representative: Dennis Wesoloski

C. Telephone Number: (312) 886-1971

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analyses for BOD5, COD, and TOC. The analysis will be performed by the Indiana State Board of Health. This SAS request is being filled out to help document the analytical protocols used.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

Analyze 50 low level groundwater and leachate samples for the parameters listed above. All samples will be unfiltered.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund, Enforcement

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment: Daily by Overnight Carrier

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached ISBH method

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Two or more sample dilutions must overlap to result in a residual of D.O. 1 mg/l and a D. O. depletion of 2 mg/l. Results for 2 dilutions should agree within 15%. Prepare a seed correction bottle, a dilution water control in duplicate and a glucose-glutamic acid check in addition to sample dilutions. Determine the initial and final D.O. of each bottle. Store samples at 4 ° C until analysis. The holding time is not to exceed 48 hours form time of sample collection. D.O. meter error is not to exceed 0.1 mg/l, 5 days apart. Use only the method specified above. The seed control sample should be run at 10 times the seed concentration. The result of the seed control samples should then be adjusted 1/10 before being used. Do not use the blank results to calculate the seed concentration. The calibration curve will include at least five standards.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Submit all raw data. Report initial and final D.O. from each bottle. Report BOD in mg/l for each bottle and the average of each dilution fitting the depletion range listed above using calculations specified by "Standard Methods". Report results of duplicates, dilution water control, seed control and glucose-glutamic acid check. All records of analysis and calculations should be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
BOD	1.5 mg/l	10% or +/- 0.5 mg/l For concentrations 5 mg/l BOD

II. QUALITY CONTROL REQUIREMENTS

Audits Required	**Frequency of Audits**	**Limits*** (+/- % or conc.)
Glucose-glutamic acid	1 per run of samples	160-240 mg/l
Duplicate	2 for runs (10	+/- 10%
Dilution Water Control	2 per batch of dilution water	(0.2mg/l
Seed control sample	2 per batch of dilution water	
EPA QC Demand Reference 1 set of 2 ampules	1 per this project	80% - 120% recovery

III. *Action Required if Limits are Exceeded:

Contact Chuck Elly at EPA Region V CPMS (phone (312) 353-9087)

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

ISBH Low level spectrophotometric method attached for COD concentrations less than 50 mg/l. ISBH Mid level spectrophotometric method attached for COD concentrations greater than 50 mg/l. Samples will be preserved in the field with 2 ml of 1:1 sulfuric acid.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Use potassium acid phthlate for the spike. Holding time is not to exceed 28 days from date of collection. The low level method will be used for COD concentrations less than 50 mg/l and the mid level method will be used for COD concentrations greater than 50 mg/l. Separate QC audits will be performed for each method if both are used. Dilute and rerun samples with absorbances higher than the highest standard.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedures used will be clearly identified. Bench records tabulating the order of calibration standards, label control standards, lab blanks, samples, etc. with resulting absorbance or concentration readouts will be provided along with copies of work sheets used to calculate results. All records of analysis and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

---

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
COD low level	5 mg/l	+/- 5 mg/l
COD mid level	50 mg/l	+/- 10 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Matrix spike [1]	2 for runs < 10 1 per 10 for runs >10	80% - 120%
Duplicate	2 for runs < 10 1 per 10 for runs >10	10% or 5 mg/l
EPA QC Demand Reference Samples [2] 1 set of 2 ampules	1 per this project	80% - 120% recovery

III. *Action Required if Limits are Exceeded:

Contact Chuck Elly at EPA Region V CPSM (phone (312) 353-9087)

1. Matrix spike will provide COD greater than 30% of the sample COD but will not exceed the working range.
2. Both the low and high level QC Demand samples will be run with the low level method but only the high level sample must be run with the high level test.

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method. Please note section 2.1 of method for types of instrumentation. Samples will be preserved with 2 ml of 50% H_2SO_4 per liter of sample and stored at 4 ° C until analysis and validation of results.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Homogenize samples if necessary. Quality results where suspended solids content may affect accuracy. Instruments with syringe injection will utilize 2 injections per measurement. Inorganic carbon values will be subtracted from total carbon values or purged from solution prior to measurement. Use only the method specified above. Obtain approval of CPMs, CRL prior to use of any other method. Use a minimum 5 point standard curve (0 and 4 standards). Dilute sample and rerun if the result is higher than the highest standard.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedures and instrument will be clearly identified. Bench records tabulating the order of calibration standards, label control standards, lab blanks, samples, etc. with resulting output or concentration readouts will be provided along with copies of work sheets used to calculate results. All records of analysis and calculations must be legible. Specify the organic compound used to prepare standards and spikes.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
TOC	0.5 mg/l	+/- 10% or (0.5 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Matrix spike [1]	2 for runs (10 1 per 10 for runs)10	85% - 110%
Lab Duplicate	2 for runs (10 1 per 10 for runs)10	+/- 10% or 0.5 mg/l
Lab Blank	2 for runs (10 1 per 10 for runs)10	(0.5 mg/l
EPA QC Demand Reference 1 set of 2 ampules	1 per this project	80% - 115% recovery
Calibration verification check standard	1 at beginning of run and 1 per 10 samples	90% - 110% recovery

III. *Action Required if Limits are Exceeded:

Contact Chuck Elly at EPA Region V CPMS (phone (313) 353-9087)

1. Matrix spike will be greater than 30% of the sample but will not exceed the working range of the instrument.

U.S. Environmental Protection Agency
HWI Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[x] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V, NSL/ECC

B. Regional Representative: Dennis Wesoloski

C. Telephone Number: (312) 886-1971

D. Date of Request: _____

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analyses for alkalinity, total suspended solids, total dissolved solids, volatile suspended solids, nitrate/nitrite, ammonia, chlorides, total phosphorous, total kjeldahl nitrogen, oil and grease, and sulfates. All samples will be run by the Indiana State Board of Health. This SAS request is being filled out to help document the analytical protocols used.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

Analyze 50 low level groundwater and leachate samples for the parameters listed above. All samples will be unfiltered.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund, Enforcement

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment: Daily by Overnight Carrier

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method. ISBH Code No. Alk-B-11-81

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Samples should be stored at 4 • C until analysis and validation of results. Sample holding time should not exceed 7 days from date of collection. Use potentiometric titration to pH 4.5 for alkalinity concentrations equal to or greater than 20 mg/l as $CaCO_3$. Do not use titrant volumes greater than 50 ml. Use only the method specified above. Obtain approval of CPMs, CRL prior to use of any other method.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedures used will be clearly identified. Bench records tabulating the order of titrant standardization, lab blanks, samples, lab control standard, spikes, duplicates, etc. with resulting titrant volume or titrant readouts will be provided along with copies of work sheets used to calculate results. All records of analysis and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

ALKALINITY - page 3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Alkalinity	3 mg/l for low level and 20 mg/l high level	+/- 10% for >20 mg/l $CaCO_3$ and +/- 2 mg/l $CaCO_3$ for <20 mg/l $CaCO_3$

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Sample spike	1 per run and 1 per 20 samples	85% - 115% recovery
Lab Duplicate	1 per run and 1 per 10 samples	+/-10% for high level
Lab Blank	1 per run and 1 per 10 samples	<5 mg/l for high level <2 mg/l for low level
EPA QC Demand Reference 1 set of 2 ampules	1 per this project	80% - 115% recovery
Titrant standardization	once each week	

III. *Action Required if Limits are Exceeded:

Contact Chuck Elly at EPA Region V CPMS (phone (312) 353-9087)

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method. ISBH Code No. SNF-A-3-74 using glass fiber filter discs without organic binder such as Millipore AP-40, Reeves Angel 934-AH, Gelman A/E, or equivalent. Membrane filter apparatus using 47mm diameter glass fiber filter and coarse (40-60) micron fritted disc as filter support must be used. The filter and support specifications are mandatory. Sample will be collected in a one liter bottle and must be kept at 4 • C until data are validated. Holding time is 7 days from date of collection.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1. Do not filter more than a 200 ml sample aliquot.
2. Duplicate sample aliquots will be filtered with two or more intervening samples
3. Aliquot filtered should provide residue greater than 1.0 mg for aliquots less than 200 ml.
4. Residues are to be weighed to constant weight pursuant to Part 7.1 Method 160. Final weight is to be used for calculations.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records of tare weights, final weights, volumes filtered, order of blanks, duplicates, samples filtered will be provided along with copies of worksheets used to calculate results. Specify manufacturer type and diameter (mm) of glass fiber filter used. All records of analysis and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Suspended Solids	2 - 3 mg/l for 200 ml	(0.5 mg for duplicates

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab Duplicate	1 per run and 1 per 10 samples	+/- 0.5 mg
Lab Blank	1 per run and 1 per 10 samples	+/- 0.5 mg
EPA QC Residue Reference 1 set of 2 samples	1 per this project	5 mg/l for nominal conc.

III. *Action Required if Limits are Exceeded:

Contact Chuck Elly at EPA Region V CPMS (phone (312) 353-9087)

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method. ISBH Code No. SF-A-3-74

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Use aliquots of 100 ml; however do not use sample aliquots yielding more than 200 mg of residue. Repeat analysis if residue is greater than 200 mg, using smaller aliquot. If pH is lesss than 4.0, raise pH value of aliquot to between pH 4 and 8 using NaOH. Subtract the weight of the sodium added from the weight of the residue. Samples will be kept at 4 ° C until analysis and validation of results. For TDS, the holding time is 48 hours. Contact CPMS, CRL prior to use of any other kind of method.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedure used will be clearly identified. Bench records tabulating weights used for caluculations and to determine constant weight will be provided along with copies of work sheets used to calculate TDS results. All records and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Dissolved solids	10 mg/l	+/- 10 % for duplicates or 2 mg/l for values) 200 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab Duplicate	1 per run and 1 per 10 samples	+/- 10 mg/l
Lab Blank	1 per run and 1 per 10 samples	+/- 2 mg/l or 10%
EPA QC Mineral Reference set of 2 samples	1 per this project	85% - 115% revovery

III. *Action Required if Limits are Exceeded:

Contact Chuck Elly at EPA Region V CPMS (phone (312) 353-9087)

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached ISBH method. Holding time is 7 days from date of collection.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1. Furnace must be up to temperature before inserting sample.
2. Do not overload desiccator.
3. Continue to dry and weigh the sample until there is less than 0.5 mg difference between successive weighings.
4. Use TSS filtered residues for analysis.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records of tare weights, final weights, order of blanks, duplicates will be provided along with copies of worksheets used to calculate results. Specify manufacturer type of muffle furnace. All records of analysis and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Suspended Solids	2 - 3 mg/l for 200 ml	(0.5 mg for duplicates

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab Duplicate	1 per run and 1 per 10 samples	+/- 0.5 mg
Lab Blank	1 per run and 1 per 10 samples	+/- 0.5 mg

III. *Action Required if Limits are Exceeded:

Contact Chuck Elly at EPA Region V CPMS (phone (312) 353-9087)

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method. ISBH Code No. (N)-B-10-82

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample aliquots will be refrigerated until analysis and validation of results. Sample holding time will not exceed 28 days. Sample aliquots will be preserved with 2 ml/liter 50% H_2SO_4. Nitrate and nitrite will be reported as mg/l N. Use only method(s) specified above. Obtain approval of CPMS, CRL, prior to use of any other method. Use minimum 5 point standard curve.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedures used will be clearly identified. Bench records tabulating the order of calibration standards, lab blanks, samples, lab control standards, etc. with resulting absorbances of concentration readouts, will be provided along with copies of work sheets used to calculate results. Only one Cd column should be used for an analytical run. If the column is changed, then the system must be recalibrated and a new set of audits is required. All records of analysis and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
NO3 - NO2	0.1 mg/l as N	+/- 10 % for duplicates +/- 0.1 mg/l for <1.0 mg/l

Results will be reported to the nearest 0.05 mg/l for concentrations <1.0 mg/l and to 2 significant figures for concentrations >1 mg/l - N.

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab Duplicate	1 per run and 1 per 10 samples	10% of 0.1 mg/l
Lab Blank	1 per run and 1 per 10 samples	0.1 mg/l - N
EPA QC Nutrient std. 1 and 2 or 1 set of 2 EPA, GC water supply nitrate samples	1 per this project	85% - 115%
Matrix spike [1]	1 per run and 1 per 10 samples	85% - 115% recovery
Lab control check std.	1 per 10 samples and beginning of each run	85 % - 115 %

III. *Action Required if Limits are Exceeded:

Contact Jay Thakkar or Chuck Elly at the Region V CRL if limits are not met after reanalyzing the sample.

1. Sample spike concentration will be greater than 30% of the sample concentration, but spiked sample will not exceed the working range of the standard curve.

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method. ISBH Code No. NH3-A-8-81

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample aliquots will be preserved with 2 ml/l H_2SO_4. Ammonia will be reported as mg/l N. Samples will be analyzed within 28 days after collection. Use a minimum 5 point standard curve (blank and 4 standards). Obtain approval of CPMs, CRL prior to use of any other method.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Test procedure used will be clearly identified. Bench records tabulating calibration standards, lab blanks, samples, lab control standards, etc. with resulting absorbance or concentration readouts will be provided along with copies of work sheets used to calculate ammonia results. All records of analyses and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Ammonia	0.1 mg/l	+/- 10 % for duplicates or (0.1 mg/l for concentrations (1 mg/l.

Report results to the nearest 0.05 mg/l and to 2 significant figures for concentrations exceeding 1 mg/l N.

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab Duplicate	1 per run and 1 per 10 samples	+/- 10% or 0.1 mg/l
Lab Blank	1 per run and 1 per 10 samples	0.1 mg/l
EPA QC Nutrient ref. 1 set of 2 samples	1 per this project	85% - 115% recovery
Matrix spike [1]	1 per run and 1 per 10 samples	85% - 115%
Laboratory control std.	1 per 10 samples and at the end of each run	85 % - 115 %

III. *Action Required if Limits are Exceeded:

Contact Jay Thakkar or Chuck Elly at the Region V CRL if limits are not met after reanalyzing the sample.

1. Sample spike concentration will be greater than 30% of the sample concentration, but spiked sample will not exceed the working range of the

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method. ISBH Code No. C1-C-6-79

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample will be kept at 4 C until analysis and validation of results. Dilute and rerun samples with absorbances higher than the highest standard. The holding time is not to exceed 28 days from the date of sample collection. Standards will be prepared daily from the stock solution. Automated potentiometric titrators can be used for Standard Methods 407c. A minimum 5 point standard curve should be used (0 to 4 standards). Use only method specified above. Obtain approval of CPMS, CRL, prior to use of any other method. Rewrite SAS request to reflect new methodology.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify the method used. Bench records tabulating the order of titrant standardization, lab blanks, duplicates, samples, spikes, etc., with resulting titrant volumes or absorbance readings will be provided along with copies of worksheets used to calculate results. All records of analysis and calculations must be legible.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

CHLORIDES - page 3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Chlorides	1 mg/l	+/- 10 % or within 1 mg/l for conc. (10 mg/l results to be reported to the nearest 1 mg/l and to 2 significant figures for conc. exceeding 10 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab Duplicate	1 per run and 1 per 10 samples	10% or 3 mg/l
Lab Blank	1 per run and 1 per 10 samples	(3 mg/l
EPA QC Mineral ref. samples, 1 set of 2 ampules	1 per this project	85% - 115%
Matrix spike [1]	1 per run and 1 per 10 samples	85% - 115% recovery
Calibration verification check sample	1 per 10 samples and beginning of each run	90 % - 110 %

III. *Action Required if Limits are Exceeded:

Contact Jay Thakkar or Chuck Elly at the Region V CRL if limits are not met after reanalyzing the sample.

1. Sample spike concentration will be greater than 30% of the sample concentration, but spiked sample will not exceed the working range of the standard curve.1

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method. ISBH Code No. PN-A-81

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample aliquots will be preserved in the field using 1ml/liter H_2SO_4 and stored at 4 C until analysis and validation of results. Holding time is not to exceed 28 days from the time of sample collection. Use only method specified above. Obtain approval of CPMS, CRL, prior to use of any other method. Rewrite SAS request to reflect new methodology. Minimum 5 point standard curve.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify the method used. Bench records and calculations for samples, blanks, duplicates, spikes and all control checks with absorbances and concentrations will be provided with copies of the worksheets. Results to be reported as mg/l P.

10. Other (use additional sheets or attach supplementary information, as needed):

---

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Total Phosphorous	0.03 mg/l	+/- 10 % or within 0.1 mg/l for conc. for conc. (1 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab Duplicate	1 per run and 1 per 10 samples	10% or 0.1 mg/l
Lab Blank	1 per run and 1 per 10 samples	0.03 mg/l
EPA QC Nutrient ref. samples, 1 set of 2	1 per this project	90% - 110%
Matrix spike (org N)	1 per run and 1 per 10 samples	90% - 110% recovery
Calibration standard	1 per 10 samples and end of set	90 % - 110 %

III. *Action Required if Limits are Exceeded:

Contact Jay Thakkar or Chuck Elly at the Region V CRL if limits are not met after reanalyzing the sample.

1. Sample spike concentration will be greater than 30% of the sample concentration, but spiked sample will not exceed the working range of the standard curve.1

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method. ISBH Code No. TKN-B-7-82

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample aliquots will be preserved in the field using 1ml/liter sulfuric acid and should be kept at 4 C until analysis and validation of results. Report results as mg/l N. Holding time is not to exceed 28 days from the time of sample collection. Use only method specified above. Obtain approval of CPMS, CRL, prior to use of any other method. Rewrite SAS request to reflect new methodology.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify the method used. Copies of all bench records tabulating the duplicates, standards, lab blanks, lab control standard samples, sample results with absorbances and concentrations are to be reported and legible. Report results in mg/l N. Provide digestion logs showing sample aliquots and concentrations of all solutions tested.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
TKN	0.1 mg/l	+/- 10 % or within (0.1 mg/l for conc. for conc. < 1 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab Duplicate	1 per run and 1 per 10 samples	10% or 0.1 mg/l
Lab Blank	1 per run and 1 per 10 samples	< 0.1 mg/l - N
EPA QC Nutrient ref. samples, 1 set of 2	1 per this project	85% - 115%
Matrix spike (org N)	1 per run and 1 per 10 samples	85% - 115% recovery
Calibration standard	1 per 10 samples and end of set	85 % - 115 %

III. *Action Required if Limits are Exceeded:

Contact Jay Thakkar or Chuck Elly at the Region V CRL if limits are not met after reanalyzing the sample.

1. Sample spike concentration will be greater than 30% of the sample concentration, but spiked sample will not exceed the working range of the standard curve.1

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method. ISBH Code No. O.G-B-2-74

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample aliquots will be preserved with 4ml/liter 1:1 (v/v) H_2SO_4. The holding time should not exceed 28 days. Use only the method specified above. Obtain approval of CPMS, CRL, prior to use of any other method. Rewrite SAS request to reflect new methodology.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Report all raw data including notebook entries, calculations, etc. Report bench records of tare weights and sample volumes along with copies of worksheets used to calculate results.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Oil and Grease	5 mg/l	+/- 10 % or within 2.5 mg/l for conc. <25 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab duplicate	1 per group of 10 samples or less	+/-25%
Lab Blank	1 per run and 1 per 10 samples	+/- 5 mg/l
EPA QC reference mtl.	1 per this project	85% - 115%

III. *Action Required if Limits are Exceeded:

Contact Jay Thakkar or Chuck Elly at the Region V CRL if limits are not met after reanalyzing the sample.

1. Sample spike concentration will be greater than 30% of the sample concentration, but spiked sample will not exceed the working range of the standard curve.1

6. Approximate number of days results required after lab receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

See attached method. ISBH Code No. B-11-81

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Dilute and rerun samples with absorbances higher than the highest standard. The holding time is not to exceed 28 days from the date of sample collection. Standards will be prepared daily from the stock solution. Use only the method specified. Obtain approval of CPMS, CRL prior to using any other method.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify the method used. Bench records tabulating the calibration standards, lab blanks, duplicates, samples, and spikes will be provided along with copies of worksheets used to calculate results. All records of analysis and calculations must be legible. Report results in mg/l SO_4.

10. Other (use additional sheets or attach supplementary information, as needed):

---

11. Name of sampling/shipping contact: Jeff Keiser

Phone: (414) 272-2426

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

SULFATES - page 3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Sulfates	5 mg/l turbidimetric 3 mg/l methyl thymol blue	+/- 10 % for conc)20 mg/l +/- 2 mg/l for conc. (20 mg/l

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Lab Duplicate	1 per run and 1 per 10 samples	+/- 10% or 2 mg/l
Lab Blank	1 per run and 1 per 10 samples	(3 mg/l
EPA QC Mineral ref. std. 1 set of 2 samples	1 per this project	85% - 115%
Matrix spike [1]	1 per run and 1 per 10 samples	85% - 115% recovery
Continuing Calib check	1 per 10 samples and beginning of each run	90 % - 110 %

III. *Action Required if Limits are Exceeded:

Contact Jay Thakkar or Chuck Elly at the Region V CRL if limits are not met after reanalyzing the sample.

1. Sample spike concentration will be greater than 30% of the sample concentration, but spiked sample will not exceed the working range of the standard curve.

Appendix D
ISBH ANALYSES

BIOCHEMICAL OXYGEN DEMAND (BOD)
5 Days, 20° C.

ISBH Code No. BOD-A-5-77
STORET No. 00310
Approved for NPDES

1. Scope and Application

1.1 The biochemical oxygen demand is used for determining the relative oxygen requirements of municipal wastewater, industrial wastes, and surface waters.

1.2 The limit of detection is 1 mg/l and the working range is 1 to 6 mg/l (other concentration ranges obtained by dilution).

2. Summary of Method

2.1 The BOD test is an empirical bioassay-type procedure which measures the dissolved oxygen consumed by microbial life while assimilating and oxidizing the organic matter present. The standard test conditions include dark incubation at 20° C for a five day period. The reduction in dissolved oxygen concentration during the incubation period yields a measure of the biochemical oxygen demand.

3. Sample Handling and Preservation

3.1 A two quart polyethylene bottle is an acceptable container.

3.2 All samples must be cooled to 4° C until such time as the dilutions are prepared for the BOD determination.

3.3 BOD dilutions should be prepared and incubation started within 24 hours after the sample has been collected or the compositing has been completed.

4. Comments

4.1 Samples should be warmed to 20° C before analysis.

4.2 The pH of the sample should be between 6 and 8.

4.3 Residual chlorine should be removed before analysis.

4.4 Any sample with dissolved oxygen concentraton of 9.0 mg/l or more at 20° C is considered supersaturated and must be corrected before dilutions are made.

4.5 Some types of wastes (high in metals, cyanide, pesticide, or herbicide wastes) may be toxic to the microorganisms used to seed the sample dilutions. If toxicity is suspected, it should be recorded on the laboratory bench sheet, and the final report form for the sample. To obtain valid BOD results on this type of waste, the seed material used to prepare sample dilutions must be acclimated to the waste.

5. Apparatus

5.1 BOD incubator which will maintain a temperature of 20 ± 1° C and also exclude light.

5.2 YSI Model 54 Oxygen Meter, or the equivalent, dissolved oxygen probe and standard membrane kit.

5.3 Magnetic stirrer.

6. Reagents

6.1 Dissolved oxygen determination.

6.1.1 Distilled water which is free of chlorine residual.

6.1.2 Manganese sulfate solution - dissolve 364 g manganous sulfate monohydrate in 700 ml distilled water. Dilute to one liter and filter before use. This solution should not give a color with starch when added to an acidified solution of potassium iodide.

6.1.3 Alkali-iodide-azide-reagent - Add 600 g reagent grade potassium iodide (KI), 1000 ml of distilled water, 2600 ml 50% $NaOH_3$ and 40 g NaN_3. Dilute to 4 liters with distilled water. Store in a polyethylene bottle with a tight fitting cap. (Warning! This reagent is extremely caustic and may cause serious burns if splashed on skin or eyes. Sodium azide will form explosive azides with lead or copper plumbing and should be used only with plastic or glass drains and pipes.)

6.1.4 Sulfuric acid, concentrated, reagent grade.

6.1.5 Sulfuric acid solution, (1 + 9) - add 10 ml reagent grade sulfuric acid to 90 ml distilled water. Mix and cool to room temperature before use.

6.1.6 Starch solution - dissolve 2 g salicylic acid in 800 ml boiling distilled water. Add a cold water suspension of 20 g soluble potato starch and stir. After two minutes add 200 ml distilled water, boil for two minutes more, cool, and allow to settle overnight.

6.1.7 Potassium iodide, crystal or granular, reagent grade, iodate free.

6.1.8 Potassium dichromate solution, 0.0250 N. - dry primary standard grade potassium dichromate at 103° for 2 hours, then dessicate at room temperature for 1 hour. Dissolve 1.226 g potassium dichromate in 500 ml distilled water and dilute to one liter. Store in a tightly capped bottle. Prepare fresh monthly.

6.1.9 Sodium thiosulfate titrant, approximately 0.0335 N. - Add 8.3147 g of sodium thiosulfate to 500 ml distilled water, add 1.5 ml 6 N. sodium hydroxide, and dilute to 1 liter. Allow this solution to remain undisturbed for 24 hours before standardization. The procedure for standardization follows:

a. Dissolve approximately 2 g potassium iodide crystals in 150 ml distilled water. Add 1 ml of 50% sulfuric acid and then 20.00 ml potassium dichromate solution. Dilute to 100 ml, mix, and place the titration vessel in the dark for 5 minutes before titrating.

b. Titrate the solution prepared above with the sodium thiosulfate titrant until a pale straw color is reached. Add approximately 1 ml starch solution and continue the titration until the blue color just disappears. Record the volume of sodium thiosulfate titrant used.

c. Repeat steps a & b at least three times until three titrations match within 0.10 ml. Average three values.

d. Calculate the normality of the sodium thiosulfate solution:

$$N. = \frac{(0.025\ N. \times 20\ ml)}{\text{average value from step c}}$$

Standardize the titrant each week it is to be used. The date, analyst name, normality of the thiosulfate should be recorded on each BOD work sheet.

6.2 BOD determination

6.2.1 Acid solution, 1 N. - dissolve 28 ml reagent grade concentrated sulfuric acid in 500 ml distilled water. Cool to room temperature and dilute to 1 liter with distilled water.

6.2.2 Base solution, 1 N. - dissolve 40 g reagent grade sodium hydroxide in 500 ml distilled water. Cool to room temperature and dilute to 1 liter with distilled water.

6.2.3 Calcium chloride solution - dissolve 27.5 g anhydrous reagent grade calcium chloride in distilled water and dilute to one liter.

6.2.4 Distilled water - store sufficient chlorine-free distilled water in a loosely-capped, chemically clean, glass or plastic carboy (2.5 or 5 gal) in the 20° C incubator. The storage period should be no less than 20-24 hours and unused water should be dumped after one week and replaced with fresh.

6.2.5 Ferric chloride solution - dissolve 0.25 g reagent grade ferric chloride hexahydrate, in 500 ml distilled water. Dilute to one liter with distilled water.

6.2.6 Magnesium sulfate solution - dissolve 22.5 g reagent grade magnesium sulfate, heptahydrate, in 500 ml distilled water. Dilute to one liter with distilled water.

6.2.7 Phosphate buffer solution - dissolve 8.5 g reagent grade potassium hydrogen phosphate, 33.4 g disodium hydrogen phosphate heptahydrate, and 1.7 g ammonium chloride in 500 ml distilled water. Dilute to one liter with distilled water.

6.2.8 Potassium iodide solution - dissolve 10 g reagent grade potassium iodide in 100 ml distilled water. Prepare this solution only when needed.

6.2.9 Sodium sulfite solution - dissolve 1.575 g reagent grade anhydrous sodium sulfite in 500 ml distilled water. Dilute to one liter with distilled water. This solution is not stable and must be prepared weekly.

6.2.10 Sulfuric acid solution (1 N.) - dilute 29 ml reagent grade concentrated sulfuric acid in one liter distilled water.

6.3 Glucose - glutamic acid solution - dry reagent grade glucose and reagent grade glutamic acid at 103° C for one hour. Add 150 mg glucose and 150 mg glutamic acid to distilled water and dilute to one liter. Sterilize in autoclave and dispense into 100 ml storage bottles. Store in 4° C refrigerator.

7. Procedure

7.1 Standardization of the dissolved oxygen meter and probe.

7.1.1 Mix the distilled water stored for preparing BOD dilution to ensure a uniform concentration of dissolved oxygen.

7.1.2 Discard the first 300 ml of the distilled water drawn through the tygon tubing that is attached to the distilled water carboy. Using the tygon tubing, fill three 272 ml BOD bottles with a minimum of surface agitation and entrained air. Fill each bottle to over flowing and cap immediately.

7.1.3 To two of the bottles, deliver 2 ml manganese sulfate solution and then 2 ml alkali-iodide-azide reagent below the surface using a serological pipet. Cap immediately and invert the bottles at least 15 times. When the precipitation has settled (2/3 of bottle contains clear supernatant), invert again at least 15 times. Allow the precipitate to settle, then add 2 ml of concentrated sulfuric acid, stopper immediately, and invert until the floc completely dissolves. The solution should be a clear, yellowish-brown in color.

7.1.4 Place the entire solution into a 500 ml Erlenmeyer flask and titrate with standardized sodium thiosulfate solution to a pale straw color. Add 1 ml starch solution and complete the titration until the blue color just disappears. Titrate the solution in the

second BOD bottle. Use the average of the two titrations for the dissolved oxygen concentration of the distilled water.

7.1.5 Check the dissolved oxygen meter according to the manufacturer's instructions for battery charge and instrument zero. Check the probe membrane for tears, wrinkles, or bubbles. Replace the membrane and filler solution if these conditions occur, or at least once each week.

7.1.6 Establish the true zero for the meter-probe combination by placing the probe in a BOD bottle containing distilled water and an excess of reagent grade sodium sulfite. Rinse the probe with distilled water after this step has been completed.

7.1.7 Calibrate the meter-probe combination using the third BOD bottle containing distilled water that was collected in step 7.1.2 above. This calibration procedure should be performed each day the meter-probe combination is used. Calibration is good for approximately four hours.

7.1.8 Store the probe in a BOD bottle filled with distilled water.

7.2 Sample pre-treatment.

7.2.1 Temperature - Warm samples to room temperature before proceeding with the analysis.

7.2.2 pH - If the pH of the sample is not between 6 and 8, then it must be neutralized before BOD dilutions are made. The pH adjustment is made with 1 N sulfuric acid or 1 N sodium hydroxide to a pH 7.

7.2.3 Chlorinated samples - Samples should not contain residual chlorine. The following procedure should be used to detect and remove the residual chlorine before BOD analysis:

a. To a 100 ml aliquot of well mixed sample, add sufficient 2% H_2SO_4 to adjust pH to 4, add 1 scoop of potassium iodide crystals, and 1 ml starch solution. If a blue color develops, titrate with sodium sulfite solution until the blue color just disappears.

b. To a measured quantity of well mixed sample which is sufficient to prepare BOD dilutions, add sodium sulfite solution in the proportions determined in 7.2.3 a. Shake the sample to remove the residual chlorine and to help oxidize any excess sodium sulfite.

7.2.4 Supersaturation - Any water sample with a dissolved oxygen of 9 mg/l or more is considered supersaturated and it must be corrected before the BOD dilutions are made. Transfer a quantity of sample, which will be used for BOD to a clean, dry bottle. Shake the sample vigorously until the excess dissolved oxygen is removed.

7.2.5 Seed - The sample dilutions of chlorinated samples, strongly acidic or basic samples, and many industrial wastes may not contain a sufficient number of micro-organisms to produce reliable results and must be seeded with organisms by the addition of a known quantity of settled sewage to the sample dilution.

a. As a general rule, seed all sample dilutions which have been chlorinated, neutralized, or collected from industrial wastes.

b. Use seed from settled domestic wastewater that has been stored at 20° C for 24-36 hours.

7.3 Preparation of sample dilutions

7.3.1 Dilution water - To a carboy of distilled water which has been stored at 20° C (6.2.4), add 1 ml/l of each: phosphate buffer solution (6.2.7), magnesium sulfate solution (6.2.6), calcium chloride solution (5.2.3), and ferric chloride solution (6.2.5). Mix and discard the first 300 ml of this solution that is dispensed through the attached tygon tubing.

7.3.2 The number and extent of sample dilutions taken depends on the expected strength of the sample. As a rough guide, the following ranges of BOD_5 values can be expected for the types of samples shown:

Sample Type	Expected BOD (range)
suface water	0-20 mg/l
polluted surface water	10-50
sewage (treated effluent)	10-500

Sample Type	Expected BOD (range)
sewage (domestic)	100-500
industrial waste	10-500
strong industrial waste	500-5,000
slaughterhouse, dairy, and feedlot wastes (untreated)	1,000-20,000

The sample should be diluted so that a residual D.O. of at least 1 mg/l remains after 5 days incubation and the uptake of dissolved oxygen at least 2 mg/l occurs. Several dilutions of the sample are prepared to obtain dissolved oxygen uptake in this range.

7.3.3 Dilutions greater than 1:100 - Make a primary dilution of the sample in a graduated cylinder and the final dilution directly in the bottle.

Dilutions less than 1:100 - Place the volume of sample directly into the bottle and if needed, add 1 ml of seed (7.2.5) to the BOD bottle. Slowly fill the remainder of the bottle with dilution water (7.3.1) so that the insertion of the stopper displaces any possible air, leaving no bubbles.

7.4 **Seed**

7.4.1 **It is necessary to have present a population of microorganisms capable of oxidizing the biodegradable organic matter in the sample. Each sample which might be deficient in microbial population (7.2.5) must have additional seed material. This is done by placing 1 ml of seed material directly into the**

bottle before the dilution water is added and this seed should contribute between 0.6 and 1 mg/l in the oxygen uptake if the BOD of the seed is approximately 200 mg/l.

7.4.2 If the samples are seeded, a BOD must be run on the seed material and the 5 day oxygen uptake must be used to correct the seeded sample dilutions. The seed material is diluted to a proportion which will produce a residual D.O. of at least 1 mg/l and a D.O. depletion of at least 2 mg/l. An initial D.O. is obtained at the same time the sample dilutions are read, the seed dilution is incubated for 5 days, and the final D.O. is read at the same time as the samples.

7.5 Dissolved Oxygen (D.O.) readings and sample incubation

7.5.1 The initial dissolved oxygen is read on each sample dilution by the membrane electrode method. Any sample volume which has been lost in reading the dissolved oxygen should be replaced with dilution water.

7.5.2 The sample is stoppered tightly and incubated for 5 days at 20° C. The water seal which is required during incubation is obtained by inverting the BOD bottle in a pan which contains water.

7.5.3 After a 5 day incubation period, the final dissolved oxygen reading is obtained for all sample dilutions.

7.6 Dilution water blank

Use dilution water blanks as a rough check on the quality of the unseeded dilution water and the cleanliness of the incubation bottles. When making initial sample dilutions, the first and last BOD bottle should be used as dilution water blanks. Intermittant blanks should be used if the number of samples is large or there is a change in the dilution water bottles. Initial D.O. readings should be taken at the same time as the sample dilutions are read (7.4.1) and final D.O. readings are taken after the 5 day incubation (7.4.2). The D.O. uptake should not be greater than 0.2 mg/l or there is a problem in the quality of the dilution water.

7.7 Glucose - glutamic acid check

7.7.1 A mixture of glucose-glutamic acid is analyzed for BOD with each sample run. The measurement of the pure organic compounds will give an indication of the dilution water quality, seed effectiveness, and the analytical technique.

7.7.2 A 1.45% and 2.54% solution of the glucose-glutamic acid solution (6.3) is made, seed added, and the 5 day BOD is obtained as outlined in 7.4. If the 5 day BOD value of the check is outside 200 ± 37 mg/l, reject any BOD determinations made with the seed and dilution water and seek the cause of the problem.

8. Calculations

8.1 The sample description, lab number, date and values for D.O. readings, and dilutions are recorded on the bench sheet. (Attachment 1)

8.2 Calculations of the 5 day BOD for samples not seeded, the seed dilution, and the dilution water blank:

$$BOD_5 = D_1 - D_2/P$$

D_1 = initial D.O. reading

D_2 = final D.O. reading after 5 day incubation

P = decimal fraction of the sample used to make the sample dilution.

8.3 Calculation of 5 day BOD for the glucose-glutamic acid dilution and the seeded sample:

$$BOD_5 = [(D_1 - D_2) - (B_2 - B_1)\ F]/P$$

P and $D_1 - D_2$ defined in 8.2

$B_2 - B_1$ = depletion of D.O. of the seed for 5 days

$$F = \frac{\text{\% seed in sample dilution}}{\text{\% seed in seed dilution}}$$

9. Quality Control

9.1 Internal Quality Control

9.1.1 Glucose-glutamic acid solutions (1.45% and 2.54%) are analyzed for 5 day BOD. The results are collected, treated statistically, and control limits are determined. The control limits are evaluated with each analytical run.

9.1.2 The blank dilution water is analyzed for the 5 day period. This shows the presence of organic contamination in the system. These data are collected and treated statistically to provide control information.

9.1.3 Duplicate field samples and duplicate lab samples are analyzed and the data is collected for statistical

evaluation. Control limits are placed on the analyses to provide adequate precision in the test.

9.2 External Quality Control

9.2.1 Regular participation in annual inter-laboratory audits are sponsored by USEPA, Region V. These are audits on the performance of the 5 day BOD procedure.

10. References

10.1 Standard Methods for the Examination of Water and Wastewater, 15th Edition, 1980, pp. 388-399, pp. 483-489.

10.2 Chemical Analyses for Water Quality, Training Course Manual, U.S. Department of the Interior, Federal Water Pollution Control Administration, pp. 6-1 to pp. 8-13.

CHEMICAL OXYGEN DEMAND
(Low COD Value Ampule Method)
Spectrophotometric

ISBH Code No. COD-B-12-85
STORET No. 00335
Approved for NPDES

1. Scope and Application

1.1 Chemical Oxygen Demand (COD) determines the quantity of oxygen required for oxidation of the organic matter in a water sample under controlled conditions of oxidizing agents, temperature and time.

1.2 The method is applicable to samples containing COD valves of 5-50 mg/l. Higher COD valves can be measured by the "Standard Ampule Method."

2. Summary of Method

Organic and inorganic compounds are oxidized in a sealed 10 ml expendable ampule which contains premixed COD reagents. The COD is determined using a spectrophotometer at 440 nm by measuring the concentration of the Cr(VI) ion. The path length of the ampules is 2.0 cm.

3. Sampling and Preservation

3.1 Collect the samples in glass bottles. Plastic containers can be used if there is known to be no contaminants in the containers.

3.2 Biologically active samples should be tested as soon as possible. Samples containing settleable material should be well mixed, preferably homogenized, to permit removal of represenative aliquots.

3.3 Samples may be preserved with sulfuric acid at a rate of 2 ml of 50% H_2SO_4 per liter of sample.

3.4 Store ampules in light-proof containers. Shelf life of these ampules is nine (9) months if stored in the dark.

4. Interferences

4.1 Any contamination of the sample with organic matter will cause an error in the analysis.

4.1.1 Extreme care should be exercised to avoid inclusion of organic matter in the distilled water used for reagent preparation or sample dilution.

4.2.2 Any glassware used in sample preparation should be free from organic matter.

4.2 Volatile materials may be lost if the sample is mixed with reagents prior to sealing the ampule.

4.3 Chlorides are stoichiometrically oxidized by dichromate and will give high COD valves. Sufficient mercuric sulfate is added to the reagents to complex up to 2,000 mg/l chlorides before it reacts with the dichromate. If chlorides are present in excess of 2,000 mg/l use the Titrimetric dichromate reflux method.

4.4 Spectrophotometric interferences will erroneously cause high COD valves to be determined. Although such interferences are not typically encountered, samples which might contain spectrophotometric interferences should be checked against titration to establish comparability.

5. Apparatus

5.1 Sealed 10 ml ampules which contain all reagents for the test. The ampule has two breakpoints. The second breakpoint is provided for analysis by wet chemistry methods.

5.2 Mechanical ampule sealer.

5.3 Oven or similar device capable of maintaining 150°C ± 2°.

5.4 Spectrophotometer.

6. Reagents

6.1 All reagents and catalysts are contained in the COD ampules. Proportionally, the reagents, catalyst and sample are the same as outlined in "Standard Method's" COD procedure.

6.2 COD stock standard solution: To obtain a 1,000 mg/l COD stock solution, add 0.8503 g. of potassium acid pathalate to a 1 liter volumetric flask and dilute to 1 liter. This stock is good for several months if refrigerated. The stock solution is then diluted to obtain appropriate standards for preparing the standard curve. A procedural blank on the distilled water should be run with the standards to zero the spectrophotometer. After establishing the curve, standards should be run with the samples as required to check accuracy.

6.3 Potassium acid phthalate: $C_8H_5KO_4$.

7. Procedure

7.1 Unseal the ampules by snapping the top colorbreak. Carefully add 2.5 ml of sample into each ampule such that it forms a layer on top of the reagents contained in the ampule.

NOTE: Samples containing particulates should be thoroughly homogenized and milled in a blender or similar device before adding the sample to the ampule.

7.2 Carefully seal the ampule using a mechanical ampule sealer.

7.3 Thoroughly mix the contents of the sealed ampule by shaking. CAUTION: The ampule will get very hot during mixing. It is recommended that ampules be mixed either in racks or with use of insulated gloves. Eye protection should be worn.

7.4 Place the ampules in an oven or any device capable of maintaining 150°C ± 2° for two hours. The two hour oxidation period is standard practice and is sufficient for complete oxidation of most compounds.

7.5 Mix ampule contents by shaking and allow to cool. If rapid cooling is desired, the ampules may be placed in a water bath. If, however, certain samples form crystals, discontinue the rapid cooling and allow these samples to cool slowly in the room air.

7.6 Allow any suspended precipitate to settle for ten minutes. Carefully wipe clean the ampules. Use 440 nm setting on the spectrophotometer. Use the highest standard (50 mg/l COD) to set the spectrophotometer to zero absorbance. Read the lower standards including the blank (0 mg/l COD). Since unreacted Cr(VI) is being measured, the blank will have the highest absorbance and the highest mg/l COD standard will have the lowest absorbance, thus the standard curve will slope downward to the right. By use of the standard curve, the absorbance is converted graphically into mg/l COD.

7.7 An an alternative to the spectrophotometric analysis, the second colorbreak on the ampule may be snapped and an aliquot removed from the ampule and analysis made by wet chemistry methods.

8. Calculation

If a mathematical solution for mg/l COD is required, perform the following operation:

$$\text{COD mg/l} = \frac{C_2-C_1}{A_2-A_1} \times A + 50$$

C_2 = The COD, mg/l at any point on the standard curve.

A_2 = The absorbance at the same point used for C_2.

C_1 = Any COD, mg/l, less that C_2 on the standard curve.

A_1 = The absorbance at the same point used for C_1.

A = The absorbance of the sample.

NOTE: This equation uses the 50 mg/l COD standard as zero absorbance.

9. Reference

9.1 O.I. Corporation "COD Low Level Ampule Method"
March 29, 1983

CHEMICAL OXYGEN DEMAND
(Standard Ampule Method, Spectrophotometric)

ISBH Code No. COD-A-12-85
STORET No. 00340
Approved for NPDES

1. Scope and Application

1.1 This method determines the quantity of oxygen required to oxidize the organic matter in a water sample, under controlled conditions of oxidizing agents, temperature and time.

1.2 Since the test utilizes a chemical rather than a biological process, the result has no defineable relationship to the BOD of the water sample. The test results should be considered as an independent measurement of organic matter in the sample, rather than as a substitute for the BOD test.

*1.3 The method is applicable to samples containing COD valves of 25-900 mg/1. Samples containing higher COD valves can be measured by analyzing known dilutions. Lower COD valves may be run by using the "Low COD Value Ampule Method."

2. Summary of Method

Organic and inorganic compounds are oxidized in a sealed 10 ml expendable ampule which contains premixed COD reagents. The COD is determined using a spectrophotometer at 600 nm by measuring the concentration of the Cr^{+3} ion. The path length of the ampules is 2 cm.

3. Sampling and Preservation

3.1 Collect the samples in glass bottles. Plastic containers can be used if there is known to be no contaminants in the containers.

3.2 Biologically active samples should be tested as soon as possible. Samples containing settleable material should be well mixed, preferably homogenized, to permit removal of representative aliquots.

3.3 Samples may be preserved with sulfuric acid at a rate of 2 ml of 50% H_2SO_4 per liter of sample.

3.4 Store ampules in light-proof containers. Shelf life of these ampules is nine (9) months if stored in the dark.

4. Interferences

4.1 Any contamination of the sample with organic matter will cause an error in the analysis.

4.1.1 Extreme care should be exercised to avoid inclusion of organic matter in the distilled water used for reagent preparation or sample dilution.

4.1.2 Any glassware used in sample preparation should be free from organic matter.

4.2 Volatile materials may be lost if the sample is mixed with reagents prior to sealing the ampule.

4.3 Chlorides are stoichiometrically oxidized by dichromate and will give high COD valves. Sufficient mercuric sulfate is added to the reagents to complex up to 2,000 mg/l chlorides before it reacts with the dichromate. If chlorides are present in excess of 2,000 mg/l use the titrimetric dichromate reflux method.

4.4 Spectrophotometric interferences will erroneously cause high COD valves to be determined. Although such interferences are not typically encountered, samples which might contain

spectrophotometric interferences should be checked against titration to establish comparability.

5. Apparatus

5.1 Sealed 10 ml ampules contain all reagents for the test. The ampule has two breakpoints. The second breakpoint is provided for analysis by wet chemistry methods.

5.2 Mechanical ampule sealer.

5.3 Oven or similar device capable of maintaining 150°C ± 2°.

5.4 Spectrophotometer.

6. Reagents

6.1 All reagents and catalyst are contained in the COD ampules. Proportionally, the reagents, catalyst and samples are the same as outlined in "Standard Method's" COD procedure.

6.2 COD stock standard solution: To obtain a 1,000 mg/l COD stock solution, add 0.803 g. of potassium acid phthalate to a 1 liter volumetric flask and dilute to 1 liter. This stock is good for several months if refrigerated. The stock solution is then diluted to obtain appropriate standards for preparing the standard curve. A procedural blank on the distilled water should be run with the standards to zero the spectrophotometer. After establishing the curve, standards should be run with the samples as required to check accuracy.

6.3 Potassium acid phthalate: $C_8H_5KO_4$.

7. Procedure

7.1 Unseal the ampules by snapping the top colorbreak. Carefully add 2.5 ml of sample into each ampule such that it forms a layer on top of the reagents contained in the ampule.

NOTE: Samples containing particulates should be thoroughly homogenized and milled in a blender or similar device before adding the sample to the ampule.

7.2 Carefully seal the ampule using a mechanical ampule sealer.

7.3 Thoroughly mix the contents of the sealed ampule by shaking. CAUTION: The ampule will get very hot during mixing. It is recommended that ampules be mixed either in racks or with use of insulated gloves. Eye protection should be worn.

7.4 Place the ampules in an oven or any device capable of maintaining 150°C plus or minus 2° for two hours. The two-hour oxidation period is standard practice and is sufficient for complete oxidation of most compounds.

7.5 Mix ampule contents by shaking and allow to cool. If rapid cooling is desired, the ampules may be placed in a water bath. If, however, certain samples form crystals, discontinue the rapid cooling and allow these samples to cool slowly in the room air.

7.6 Allow any suspended precipitate to settle for ten minutes. Carefully wipe clean the ampules. Read the absorbance of each ampule on a spectrophotometer set at 600 nm wavelength. Use a procedural blank run on the distilled water to set zero on the spectrophotometer. Measure the absorbance of the blank. Re-zero the instrument with the blank. By use of the standard curve (6-2), the absorbance is converted graphically into mg/l COD.

7.7 AS an alternative to spectrophotometric analysis, the second colorbreak on the ampule may be snapped, an aliquot may then

be removed from the ampule and analysis made by wet chemistry methods.

8. Calculation

If a mathematical solution for mg/l COD is required, perform the following operations:

$$\text{COD, mg/l} = \frac{C_2 - C_1}{A_2 - A_1} (A)$$

C_2 = The COD mg/l at any point on the standard curve.

A_2 = The absorbance at the same point used for C_2.

C_1 = Any COD, mg/l less than C_2 on the standard curve.

A_1 = The absorbance at the same point used for C_1.

A = The absorbance of the sample.

9. References

9.1 O.I. Corporation's "COD Standard Ampule Method" March 29, 1983.

Organic Carbon, Total
EPA Method

ISBH Code No. TOC-A-6-86
STORET No. 00680
approved for NPDES

1. Scope and Application:

   1.1 This method includes the measurement of organic carbon in drinking, surface and saline waters, domestic and industrial wastes.

   1.2 The detection limit is estimated to be about 0.3 mg/l using a 1 ml sample loop. Sample carry-over may be the main contributor to the magnitude of the detection limit.

   1.3 The working range of the method is from 0.5 to 30 mg/l with the 1 ml sample loop. Higher concentrations of T.O.C. would be in the nonlinear range and should be diluted.

2. Summary:

   2.1 Organic carbon in a sample is converted to carbon dioxide by persulfate digestion. The CO_2 formed is then measured directly by an infrared detector.

3. Sample Handling and Preservation:

   3.1 Samples are collected in 1-liter plastic or glass bottles.

   3.2 The samples are preserved with a 2 ml of 50 percent sulfuric acid per 1-liter.

4. Comments:

   4.1 Normally carbonate, bicarbonate and dissolved carbon dioxide represent an interference in T.O.C. determinations. With the O.I. Corp. Model 700 instrument, the inorganic carbon is determined separately just prior to the T.O.C.

5. Apparatus:

   5.1 O.I. Corp. Model 700 T.O.C.

   5.2 Autosampler.

   5.3 Epson RX-80 printer.

6. Reagents:

   6.1 Phosphoric acid, 5 % solution.

6.2 Sodium persulfate, 100 g/liter.

6.3 Potassium hydrogen phthalate, stock solution (1000 mg C/l): Dissolve 2.125 g. of dried potassium hydrogen phthalate in 1-liter of deionized water. Sulfuric acid is added as a preservative (2 ml 50 % H_2SO_4/l).

6.4 Working standard (25 mg/l): 25 ml of stock solution (1000 mg/l) made up to 1-liter volume and preserved with sulfuric acid.

7. Procedure:

7.1 The sample is introduced into a digestion vessel either by a syringe injection or via sample loop.

7.2 Phosphoric acid is automatically added to the sample in the digestion vessel. The CO_2 which is formed is purged by the nitrogen gas to a molecular sieve trap which is held at 25° C.

7.3 The trap is then rapidly heated to 200° C. A stream of nitrogen gas desorbs the CO_2 and carries it into the I.R. detector.

7.4 The concentration which is displayed and printed represents the total inorganic carbon (T.I.C.).

7.5 The remaining sample in the injection vessel is heated to 100° C and sodium persulfate is automatically added. The persulfate reacts with the organic carbon to produce CO_2. This is purged and trapped in the molecular sieve at 25° C.

7.6 The trap is again rapidly heated to 200° C. A stream of nitrogen gas desorbs the CO_2 and carries it into the I.R. detector.

7.7 The concentration is displayed and printed to represent the total organic carbon (T.O.C.).

7.8 The digestion vessel is then purged and rinsed with acid. The instrument is now ready for another sample.

8. Calibration:

8.1 A two point calibration is performed by analyzing reagent blanks and a 25 mg/l standard.

8.2 The values for the reagent blank and the standard are stored in the instrument's memory.

9. Quality Control:

9.1 Internal Quality Control:

M-41

9.1.1 In-House quality control standards are run with each set of samples. A record is kept of the results and statistical analyses are performed on the accumulated data. Control limits are calculated from the data and used for daily audits of the procedure.

9.1.2 Duplicates are run every 20 samples to establish the precision of the method for real sample matrices. Data are collected, statistically evaluated, and control limits are prepared. These control limits are used as daily audits for precision of the method.

9.1.3 A blank solution containing D.I. water preserved with 2 ml conc. H_2SO_4 is run periodically to check the ability of the method to reach to established detection level and to check for sample carry-over.

9.2 External Quality Control:

9.2.1 The laboratory participates in any inter-laboratory audit which is sponsored by USEPA, Region 5 or the International Joint Commission.

9.2.2. Performance Evaluation Samples are obtained from the USEPA, Region 5 to analyze periodically. The data from these samples are recorded in the quality control log book and are used for the audit of accuracy of the method.

10. References:

10.1 O.I. Corporation.
Model 700 T.O.C. Analyzer. December 1984.

Organic Carbon
F4A
7-1-86 jmw

ALKALINITY, TOTAL
(EPA Method, 1979)

ISBH Code No. Alk-B-11-81
STORET No. 00410
Approved for NPDES

1. Scope and Application

   1.1 This method is applicable to drinking waters and surface waters, domestic and industrial wastes, and saline waters.

   1.2 The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 50 ml.

2. Summary of Method

   2.1 An unaltered sample is titrated to an electrometrically determined end point of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way.

3. Sample Handling and Preservation

   3.1 The sample should be refrigerated to 4°C and run as soon as possible.

   3.2 Do not open the sample before analysis. The maximum holding time (per Methods Manual, EPA-600/4-79-020) is 24 hours.

4. Comments

   4.1 Substances such as weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.

   4.2 For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoint of pH 3.9, using the procedure in Annual Book of ASTM Standards, Part 31, "Water," p. 129, D.1067, Method D (1976).

4.3 Oil and grease, by coating the pH electrode, may interfere, causing sluggish response.

5. Apparatus

5.1 pH meter.

5.2 pH electrodes.

5.3 Magnetic stirrer, pipets, flasks, and other standard laboratory equipment.

5.4 Buret, Pyrex, 25 ml.

6. Reagents

6.1 Standard sulfuric acid, 0.02 N.

6.3 Standard sulfuric acid, 0.1 N.

7. Procedure

7.1 Sample size and Titrant

7.1.1 Use 50 ml sample or some convenient aliquot to obtain 50 ml of titrant or less.

7.1.2 For alkalinity of 1000 mg $CaCO_3$/l, use 0.02 N titrant (6.1)

7.1.3 For alkalinity of 1000 mg $CaCO_3$/l, use 0.10 N titrant (6.2)

7.2 Potentiometric titration

7.2.1 Place sample in a 150 ml beaker by pipetting with pipet tip near the bottom of the beaker.

7.2.2 Measure pH of sample.

7.2.3 Add standard acid (6.1 or 6.2), being careful to stir thoroughly but gently to allow needle to obtain equilibrium.

7.2.4 Titrate to pH 4.5 and record volume.

8. Calculations

8.1 Alkalinity, as mg/l $CaCO_3$ $= \frac{A \times N \times 50,000}{ml \text{ of sample}}$

Where: A = ml standard acid.

B = normality of standard acid

9. Precision

9.1 One of every 20 samples is run in duplicate for use as precision data.

10. References

10.1 "Standard Methods for the Examination of Water and Wastewater," 14th Edition, p. 278, Method 403 (4d) (1975).

10.2 Annual Book of ASTM Standards, Part 31, "Water," p. 129, D.1067, Method E (1976).

10.3 "Methods for Chemical Analysis of Water and Wastes," 1979, EPA-600/4-79-020.

lgf 12/2/81
W&S disk (452)/Job K

SOLIDS, NON-FILTERABLE (SUSPENDED)
(EPA Method, 1971)

ISBH Code No. SNF-A-3-74
STORET No. 00530
Approved for NPDES

1. Scope and Application

1.1 This method is applicable to surface waters, domestic and industrial wastes, and saline waters.

1.2 The practical range of the determination is 10 mg/l to 20,000 mg/l.

2. Summary of Method

2.1 A well-mixed sample is filtered through a standard glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105°C.

3. Definitions

3.1 Non-filterable solids are defined as those solids which are retained by a standard glass fiber filter and dried to constant weight at 103-105°C.

4. Sample Handling and Preservation

4.1 Non-homogenous particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample.

4.2 Preservation of the sample is not practical; analysis should begin as soon as possible.

5. Interferences

5.1 Too much residue on the filter will entrap water and may require prolonged drying.

6. Apparatus

6.1 Glass fiber filter discs, 4.7 cm or 2.2 cm, without organic binder, Reeve Angel type 984 H, Gelman type A, or equivalent.

6.2 Filter holder, membrane filter funnel or Gooch crucible adapter.

6.3 Suction flask, 500 ml.

6.4 Gooch crucibles, 25 ml (if 2.2 cm filter is used).

6.5 Drying oven, 103-105°C.

6.6 Desiccator.

6.7 Analytical balance, 200 g capacity, capable of weighing to 0.1 mg.

7. Procedure

7.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vaccuum after water has passed through. Remove filter from membrane filter apparatus or both crucible and filter if Gooch crucible is used, and dry in an oven at 103-105°C for one hour. Remove to desiccator and store until needed. Weigh immediately before use.

7.2 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 ml to the funnel by means of a 100 ml volumetric cyclinder. If suspended matter is low, a larger volume may be filtered.

7.3 Carefully remove the filter from the membrane filter assembly. Alternatively, remove crucible and filter from crucible adapter. Place in drying oven and dry at 103-105°C to constant weight.

8. Calculations

8.1 Calculate non-filterable solids as follows:

$$\text{Non-Filterable Solids mg/l} = \frac{(\text{wt. of filter + residue})_{(mg)} - (\text{wt. of filter})_{(mg)} \times 10^3}{\text{ml of sample filtered}}$$

9. References

9.1 "Standard Methods for the Examination of Water and Wastewater", 13th Edition, p. 537, Method 224C.

9.2 "Methods for Chemical Analysis of Water and Wastes", 1971, Environmental Protection Agency, p. 278.

9.3 Federal Register, Vol. 38, No. 199, (October 16, 1973), Part II, EPA, Water Programs.

SOLIDS, FILTERABLE (DISSOLVED)
(EPA Method, 1971)

ISBH Code No. SF-A-3-74
STORET No. 70300 (180°C)
00515 (105°C)
Approved for NPDES

1. Scope and Application

1.1 This method is applicable to surface waters, domestic and industrial wastes, and saline waters.

1.2 The practical range of the determination is 10 mg/l to 20,000 mg/l.

2. Summary of Method

2.1 A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C.

3. Definitions

3.1 Filterable solids are defined as those solids capable of passing through a standard glass fiber filter and dried to constant weight at 180°C.

4. Sample Handling and Preservation

4.1 Samples should be analyzed as soon as practicable.

5. Interferences

5.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying and desiccation and quick weighing.

5.2 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate.

5.3 Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying.

(Solids, Filterable)

Total residue should be limited to about 200 mg.

6. Apparatus

6.1 Glass fiber filter, 4.7 cm or 2.2 cm, without organic binder, Reeve Angel type 984 H, Gelman type A, or equivalent.

6.2 Filter holder, membrane filter funnel or Gooch crucible adapter.

6.3 Suction flask, 500 ml.

6.4 Gooch crucibles, 25 ml (if 2.2 cm filter is used).

6.5 Evaporating dishes, porcelain, 100 ml volume. (Vycor or platinum dishes may be substituted).

6.6 Steam bath.

6.7 Drying oven, 180°C±2°C.

6.8 Dessiccator.

6.9 Analytical balance, 200 g capacity, capable of weighing to 0.1 mg.

7. Procedure

7.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus or both crucible and filter if Gooch crucible is used, and dry in an oven at 103-105°C for one hour. Remove to dessiccator and store until needed.

7.2 Preparation of evaporating dishes: Heat the clean dish to 550°C

for one hour in a muffle furnace. Cool in desiccator and store until needed. Weigh immediately before use.

7.3 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 ml to the funnel by means of a 100 ml volumetric cyclinder. If suspended matter is low, a larger volume may be filtered.

7.4 Filter the sample through the glass fiber filter and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

7.5 Transfer 100 ml (or a larger volume) of the filtrate to a weighed evaporating dish and evaporate to dryness on a steam bath.

7.6 Dry the evaporated sample for at least one hour at 180°C±2°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5 mg.

7.7 Note: The filtrate from the test for SOLIDS, NON-FILTERABLE, may be used for this determination.

8. Calculation

8.1 Calculate filterable solids as follows:

$$\text{Filt. solids, mg/l} = \frac{(\text{Wt. of dried residue+dish})-(\text{wt. of dish}) \times 100}{\text{Volume of filtrate used}}$$

9. References

9.1 "Standard Methods for the Examination of Water and Wastewater", 13th Edition, p. 539, Method 224E

(Solids, Filterable)

9.2 "Methods for Chemical Analysis of Water and Wastes", 1971, Environmental Protection Agency, p. 275.

9.3 Federal Register, Vol. 38, No. 199, (October 16, 1973), Part II, EPA, Water Programs

April 1987

Indiana State Board of Health

SOLIDS, VOLATILE

Bureau of Laboratories

Environmental Laboratory Division

1330 West Michigan Street

Indianapolis, IN 46206

SOLIDS, VOLATILE
(EPA Method, 1983)

ISBH Code No. SV-A-4-87
STORET No. 00505 (ST)
00520 (SF)
00535 (SNF)
Approved for NPDES

1. Scope and Application

1.1 This method determines the weight of solid material combustible at 550° C.

1.2 The test is useful in obtaining a rough approximation of the amount of organic matter present in the solid fraction of sewage, activated sludge, industrial wastes, or bottom sediments.

2. Summary of Method

2.1 The residue obtained from the determination of total, suspended, or dissolved solids is ignited at 550° C. in a muffle furnace. The loss of weight on ignition is reported as mg/l volatile solids.

3. Comments

3.1 The test is subject to many errors due to loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion.

3.2 The results should not be considered an accurate measure of organic carbon in the sample, but may be useful in the control of plant operations.

3.3 The principal source of error in the determination is failure to obtain a representative sample.

4. References

4.1 Standard Methods for the Examination of Water and Wastewater, 16th Ed., p. 97, Method 209D.

4.2 Methods for Chemical Analysis of Water and Wastes, 1983, Environmental Protection Agency, p. 160.4.

4.3 Federal Register, Vol. 49, No. 209, (October 26, 1984), Part VIII, EPA, Water Programs.

Indiana State Board of Health
Bureau of Laboratories
Environmental Laboratory Division

April 1987

NITROGEN, NITRATE + NITRITE
Colorimetric, Automated Cadmium Reduction
ISBH Modifications to EPA Method, 1979

ISBH Code No. NO_3+NO_2(N)-B-10-82
STORET No. Total 00630
Approved for NPDES and SDWA

1. Scope and Application

1.1 This method pertains to the determinations of nitrite singly, or nitrite and nitrate combined in surface and saline waters, and domestic and industrial wastes. The applicable range of this method is 0.1 to 10.0 mg/l nitrate+nitrite nitrogen. The range may be extended with sample dilution.

2. Summary of Method

2.1 A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically. Separate, rather than combined nitrate+nitrite, values are readily obtained by carrying out the procedure first with, and then without the Cu-Cd reduction step.

3. Sample Handling and Preservation

3.1 Analysis should be made as soon as possible. If analysis can be made within 24 hours, the sample should be preserved by refrigeration at 4° C. When samples must be stored for more than 24 hours, they should be preserved with sulfuric acid (2 ml 50% H_2SO_4 per liter) and refrigeration.

Caution: Samples for reduction column must be preserved with mercuric chloride.

4. Interferences

4.1 Build up of suspended matter in the reduction column will restrict sample flow. Since nitrate-nitrogen is found in a soluble state, the sample may be pre-filtered.

4.2 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with an organic solvent.

5. Apparatus

5.1 Technicon AutoAnalyzer (AAII) consisting of the following components:

5.1.1 Sampler

5.1.2 Analytical Cartridge (AAII)

5.1.3 Proportioning Pump

5.1.4 Colorimeter equipped with a 15 mm tubular flow cell and 520 nm filters.

5.1.5 Recorder.

5.1.6 A/D Converter and Computer.

6. Reagents

6.1 Granulated cadmium: 20 mesh MCB Reagents.

6.2 Cu - Cd column:

6.2.1 The cadmium granules (new or used) are cleaned with 50% reagent grade HCl and then rinsed with distilled water. The color of the cadmium so treated should be silver.

6.2.2 Swirl approximately 10g cadmium in 10 ml aliquots of 2% $CuSO_4$.5 H_2O for at least ten 30 sec. periods.

6.2.3 Wash the cadmium with distilled water (at least 10 times) to remove the precipitated copper. The color of the cadmium should be black.

6.3 Preparation of reduction column AAII: The reduction column is a U-shaped, 35 cm length, 2 mm I.D. glass tube (Note 1). Fill the reduction column with distilled water to prevent entrapment of air bubbles during the filling operations. Transfer the copper-cadmium granules (6.2) to the reduction column and place a glass wool plug in each end. To prevent entrapment of air bubbles in the reduction column be sure that all pump tubes are filled with reagents before putting the column into the analytical system.

NOTE 1: A 0.081 I.D. pump tube (purple) can be used in place of the 2 mm glass tube.

6.4 Distilled water: Because of possible contamination, this should be prepared by passage through an ion exchange column comprised of a mixture of both strongly acidic-cation and strongly basic-anion exchange resins. The regeneration of the ion exchange column should be carried out according to the manufacturer's instructions.

6.5 Color reagent: To approximately 100 ml of distilled water, add, while stirring, 40g sulfanilamide, 2.0g N-1- Naphthyl-ethylenediamine-dihydrochloride, and 100ml concentrated phosphoric acid. Stir until dissolved and dilute to 1 liter.

6.6 Dilute hydrochloric acid, 1-N: Dilute 8.3 ml of conc. HCl to 100 ml of distilled water.

6.7 Copper sulfate solution, 2%: Dissolve 20 g of $CuSO_4.5H_2O$ in 500 ml of distilled water and dilute to 1 liter. (Making the 2% $CuSO_4$ soln. slightly acidic by the addition of HCl improves the copper coating process.)

6.8 Wash solution: Use distilled water for unpreserved samples. For samples preserved with H_2SO_4, use 2 ml 50% H_2SO_4 per liter of wash water. (Wash water is also used as dilution water.)

6.9 Ammonium chloride soln: Dissolve 85g of reagent grade ammonium chloride in 100ml of distilled water. Add 0.5 ml Brj -35 and dilute to 1 liter.

6.10 Stock nitrate solution: Dissolve 7.218 g KNO_3 and dilute to 1 liter in a volumetric flask with distilled water. Preserve with 2 ml of 50% H_2SO_4 per liter. Solution is stable for 6 months. 1 ml = 1.0 mg NO_3-N.

6.11 Stock nitrite solution: Dissolve 6.072 g KNO_2 in 500 ml of distilled water and dilute to 1 liter in a volumetric flask. Preserve with 2 ml of chloroform and keep under refrigeration. 1.0 ml = 1.0 mg NO_2-N.

6.12 Standard nitrate solution: Dilute 10ml of stock nitrate solution (6.10) to 100ml using distilled water. 1ml = 100ug NO_3-N

6.13 Standard nitrite solution: Dilute 10.0 ml of stock nitrite (6.11) solution to 1000 ml 1.0 ml = 0.01 mg NO_2-N. Solution is unstable; prepare as required.

6.14 Working standards: Using the standard nitrate solution (6.12), prepare the following standards in volumetric flasks:

Conc. mg NO_3-N/l	ml std. soln/Vol DW
0.1	1 ml/l
0.5	1/200 ml
2.0	4/200 ml
5.0	10/200 ml
7.0	14/200 ml
10.0	20/200 ml

6.15 Sodium hydroxide solution, 0.5%: Add 10ml of 50% sodium hydroxide to 500ml distilled water and dilute to 1 liter. Make fresh daily!

7. Procedure

7.1 If the pH of the sample is below 5 or above 9, adjust to between 5 and 9 with either conc. Hcl or conc. NH_4OH.

7.2 Set up the manifold as shown in Figure 1 (AAII). Care should be taken not to introduce air into reduction column on the AAII.

7.3 Allow both colorimeter and recorder to warm up for 30 minutes. Obtain a stable baseline with all reagents feeding distilled water through the sample line.

Note 3: Condition column by running 10 mg/l standard for 30 minutes if a new reduction column is being used. Subsequently wash the column with reagents for 20 minutes.

7.4 Place appropriate nitrate and/or nitrite standards in sampler in order of increasing concentration of nitrogen. Complete loading of sampler tray with unknown samples.

7.5 For the AAII, use a 40/hr., 2:1 cam.

7.6. Switch the sample line to sampler and start analysis.

7.7 After analysis, remove reduction column before cleaning the system.

7.8 For low level nitrate analysis, use standards of 2.0, 1.5, 1.0, 0.5, 0.1 mg/l. Disconnect the dilution loop and keep the remaining manifold unchanged.

8. Calculations

8.1 The AutoAnalyzers are connected to a computer which receives the response signal from the colorimeter. After the type of curve fit is selected by the operator, the computer calculates the calibration curve by least squares method and generates concentration values for the samples, quality control solution, and laboratory blanks.

8.2 The response signal from the colorimeter is also connected to a strip chart recorder. The chart can be used to calculate concentration values by use of the overlay. The standard curve is prepared on the overlay by plotting the peak heights of standards against known concentrations. The concentration of the samples are obtained by comparing sample peak heights with the standard curve. The standard curve is not linear throughout the working range.

Bibliography

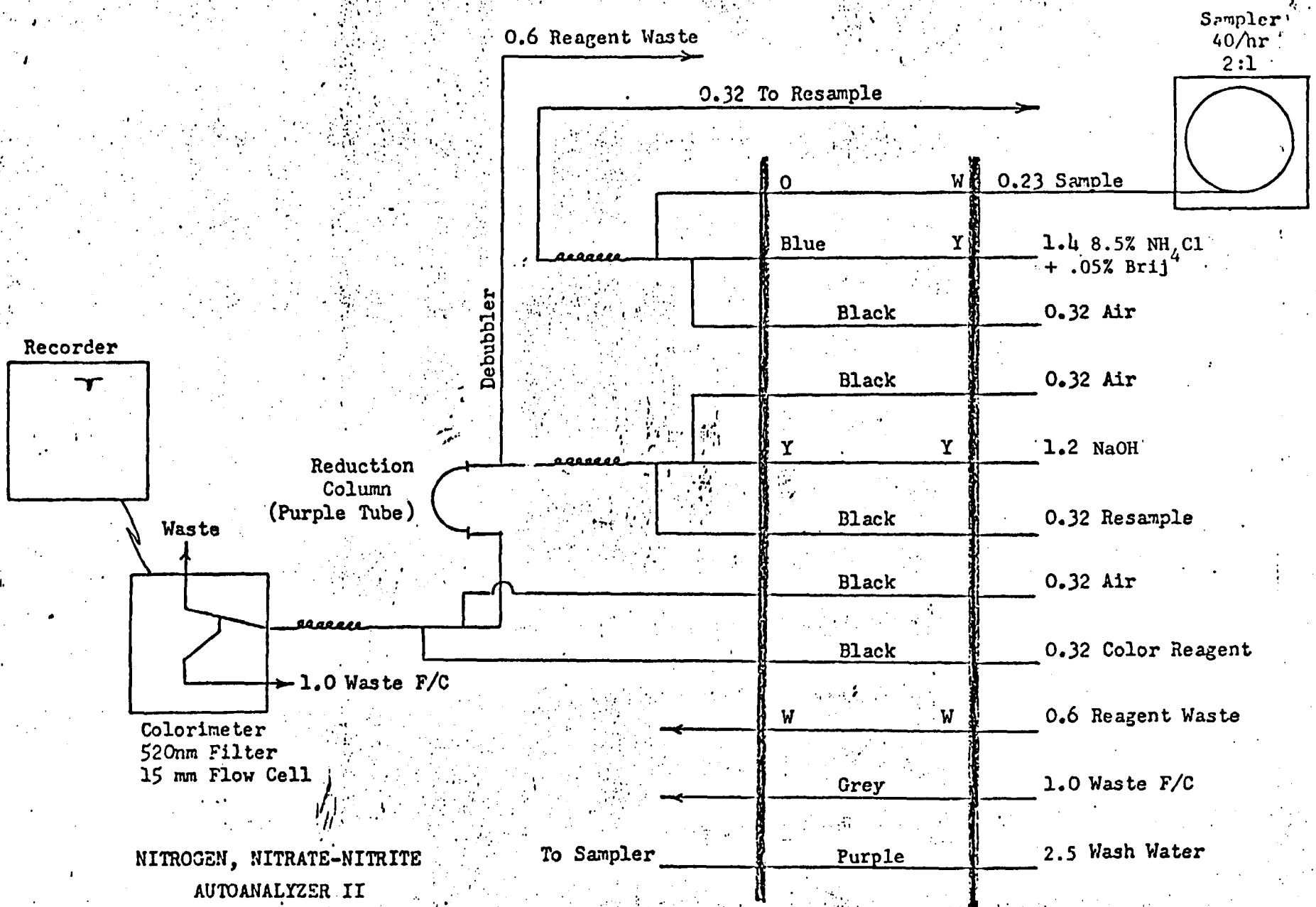
1. Fiore, J., and O'Brian, J.D., "Automation in Sanitary Chemistry-parts 1 and 2, Determination of Nitrates and Nitrites," Wastes Engineering 33, 128 & 238 (1962).

2. Armstrong, F.A., Stearns, C.R., and Strickland, J.D., "The Measurement of Upwelling and Equipment," Deep Sea Research 14, p 381-389 (1967).

3. Annual Book of ASTM Standards, Part 31, "Water," Standard D1254, p. 366 (1976).

4. Chemical Analyses for Water Quality Manual, Department of the Interior, FWPCA, R. A. Taft Sanitary Engineering Center Training Program, Cincinnati, Ohio 45226 (January, 1966).

5. Annual Book of ASTM Standards, Part 31, "Water," Standard D1141-75 Substitute Ocean Water, p 48 (1976).



NITROGEN, NITRATE-NITRITE
AUTOANALYZER II

(Modified 12-15-81)

FIGURE 1

NITROGEN, AMMONIA
Colorimetric, Automated Phenate
(ISBH Modifications to EPA Method, 1979)

ISBH Code No. NH_3-A-8-81
STORET NO. Total 00610
Approved for NPDES

1. Scope and Application

1.1 This method covers the determination of ammonia in drinking, surface, and saline waters, domestic and industrial wastes in the range of 0.10 to 10 mg/l NH_3 as N. This range is for photometric measurements made at 630-660 nm in a 15 mm or 50 mm tubular flow cell. Higher concentrations can be determined by sample dilution. Approximately 20 to 60 samples per hour can be analyzed.

2. Summary of Methods

2.1 Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside.

3. Sample Handling and Preservation

3.1 Preservation by addition of 2 ml conc. H_2SO_4 per liter and refrigeration at 4° C.

4. Comments

4.1 Calcium and magnesium ions may be present in concentration sufficient to cause precipitation problems during analysis. A sodium potassium tartrate solution is used to prevent the precipitation of calcium and magnesium ions from river water and industrial waste.

4.2 Sample turbidity and color may interfere with this method. Turbidity must be removed by filtration prior to analysis.

Sample color that absorbs in the photometric range used will also interfere.

5. Apparatus

5.1 Technicon AutoAnalyzer Unit (AAII) consisting of:

5.1.1 Sampler.

5.1.2 Analytical Cartidge (AAII).

5.1.3 Proportioning pump.

5.1.4 Heating bath with double delay coil (AAI).

5.1.5 Colorimeter equipped with 15 mm tubular flow cell and 630 nm filters.

5.1.6 Recorder.

6. Reagents

6.1 Distilled water: Special precaution must be taken to insure that distilled water is free of ammonia. Such water is prepared by passage of distilled water through an ion exchange column comprised of a mixture of both strongly acidic cation and strongly basic anion exchange resins. The regeneration of the ion exchange column should be carried out according to the instruction of the manufacturer.

NOTE 1: all solutions must be made using ammonia-free water.

6.2 Sulfuric acid: 50% sulfuric acid.

6.3 Sodium phenolate: Using a 1 liter Erlenmeyer flask, dissolve 80 ml phenol in 500 ml of distilled water. In small increments, cautiously add with agitation, 40 ml of 50% NaOH. Periodically cool flask under water faucet. When cool, dilute to 1 liter with distilled water.

6.4 Sodium hypochlorite solution: Dilute 125 ml of a bleach solution containing 5.25% NaOCl to 250 ml with distilled water. Make fresh daily!

6.5 (Replace sodium potassium tartrate solution with the following EDTA reagent)

Disodium ethylenediamine-tetraacetate (EDTA)(5%): Dissolve 50g of EDTA (disodium salt) and 20 ml 50% sodium hydroxide in 1 liter of distilled water. Add 6 drops of Brij 35.

6.6 Sodium nitroprusside (0.05%): Dissolve 0.5 g of sodium nitroprusside in 1 liter of distilled water.

6.7 Stock solution: Dissolve 3.819 g of anhydrous ammonium chloride, NH_4Cl, dried at 105°C, in distilled water, and dilute to 1000 ml 1.0 ml = 1.0 mg NH_3-N.

6.8 Standard Solution: Dilute 10.0 ml of stock solution (6.7) to 100 ml with distilled water. 1.0 ml = 0.10 mg NH_3-N.

6.9 Using standard solution, prepare the following standards:

NH_3-N, mg/l	ml Standard Solution Vol D.W.
0.1	1/1000 ml
0.5	1/200 ml
2.0	4/200 ml
5.0	10/200 ml
7.5	15/200 ml
10.0	20/200 ml

NOTE 2: When saline water samples are analyzed, Substitute Ocean Water (SOW) should be used for preparing the above standards used for the calibration curve; otherwise, distilled water is used. If SOW is used, subtract its blank background response from the standards before preparing the standard curve.

Substitute Ocean Water (SOW)

NaCl	24.53 g/l	$NaHCO_3$	0.20 g/l
$MgCl_2$	5.20 g/l	KBr	0.10 g/l
Na_2SO_4	4.09 g/l	H_3BO_3	0.03 g/l
$CaCl_2$	1.16 g/l	$SrCl_2$	0.03 g/l
KCl	0.70 g/l	NaF	0.003 g/l

6.10 The working standards for low level nitrate analysis are 2.0, 1.5, 1.0, 0.5, 0.1mg NH_3-N/l. The only modification of the manifold is disconnecting the dilution loop.

6.11 Wash water (dilution water): Add 2ml 50% sulfuric acid to 1 liter of distilled water and mix.

7. Procedure

7.1 Since the intensity of the color used to quantify the concentration is pH dependent, the acid concentration of the wash water and the standard ammonia solutions should approximate that of the samples. For example, if the samples have been preserved with 2 ml 50% H_2SO_4/liter.

7.2 For a working range of 0.1 to 10. mg NH_3-N/l (AAII), set up the manifold as shown in figure 2. Higher concentrations may be accommodated by sample dilution.

7.3 Allow both colorimeter and recorder to warm up for 30 minutes. Obtain a stable baseline with all reagents, feeding distilled water through sample line.

7.4 For the AAII use a 40/hr 2:1 cam with a common wash.

7.5 Arrange ammonia standards in sampler in order of increasing concentration of nitrogen. Complete loading of sampler tray with unknown samples.

8. Calculations

8.1 The AutoAnalyzers are connected to a computer which receives the response signal from the colorimeter. After the type of curve fit is selected by the operator, the computer calculates the calibration curve by least squares method and generates concentration values for the samples, quality control solution, and laboratory blanks.

8.2 The response signal from the colorimeter is also connected to a strip chart recorder. The chart can be used to calculate concentration values by use of the overlay. The standard curve is prepared on the overlay by plotting the peak heights of standards against known concentrations. The concentration of the samples are obtained by comparing sample peak heights with the standard curve. The standard curve is not linear throughout the working range.

Bibliography

1. Hiller, A., and Van Slyke, D., "Determination of Ammonia in Blood," J. Biol. Chem. 102, p 499 (1933).

2. O'Conner, B., Dobbs, R., Villiers, B., Dean, R., "Laboratory Distillation of Municipal Waste Effluents," JWPCF 39, R 25 (1967).

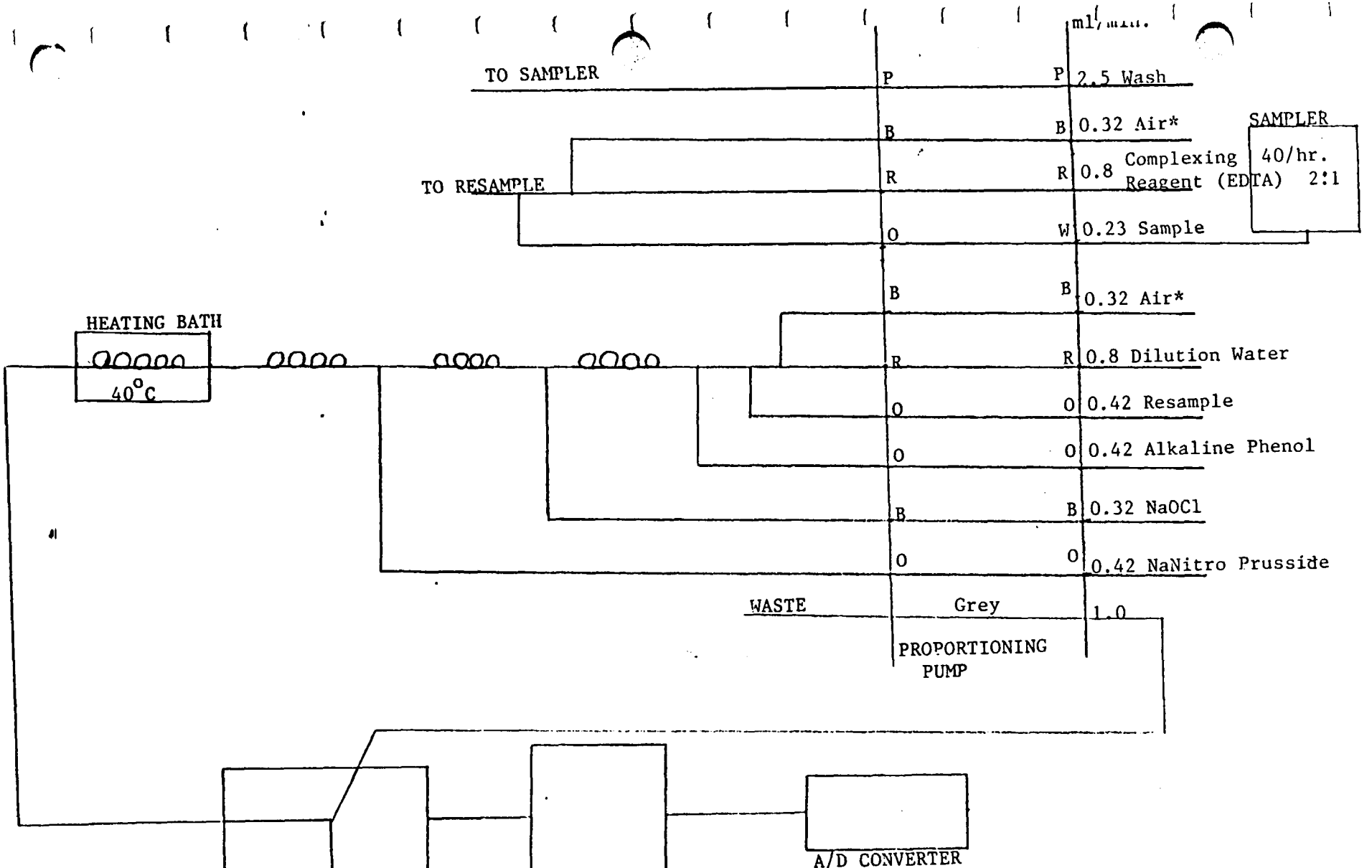
3. Fiore, J., and O'Brien, J. E., "Ammonia Determination by Automatic Analysis," Wastes Engineering 33, p 352 (1962).

4. A wetting agent recommended and supplied by the Technicon Corporation for use in AutoAnalyzers.

5. ASTM "Manual on Industrial Water and Industrial Waste Water," 2nd Ed., 1966 printing, p 418.

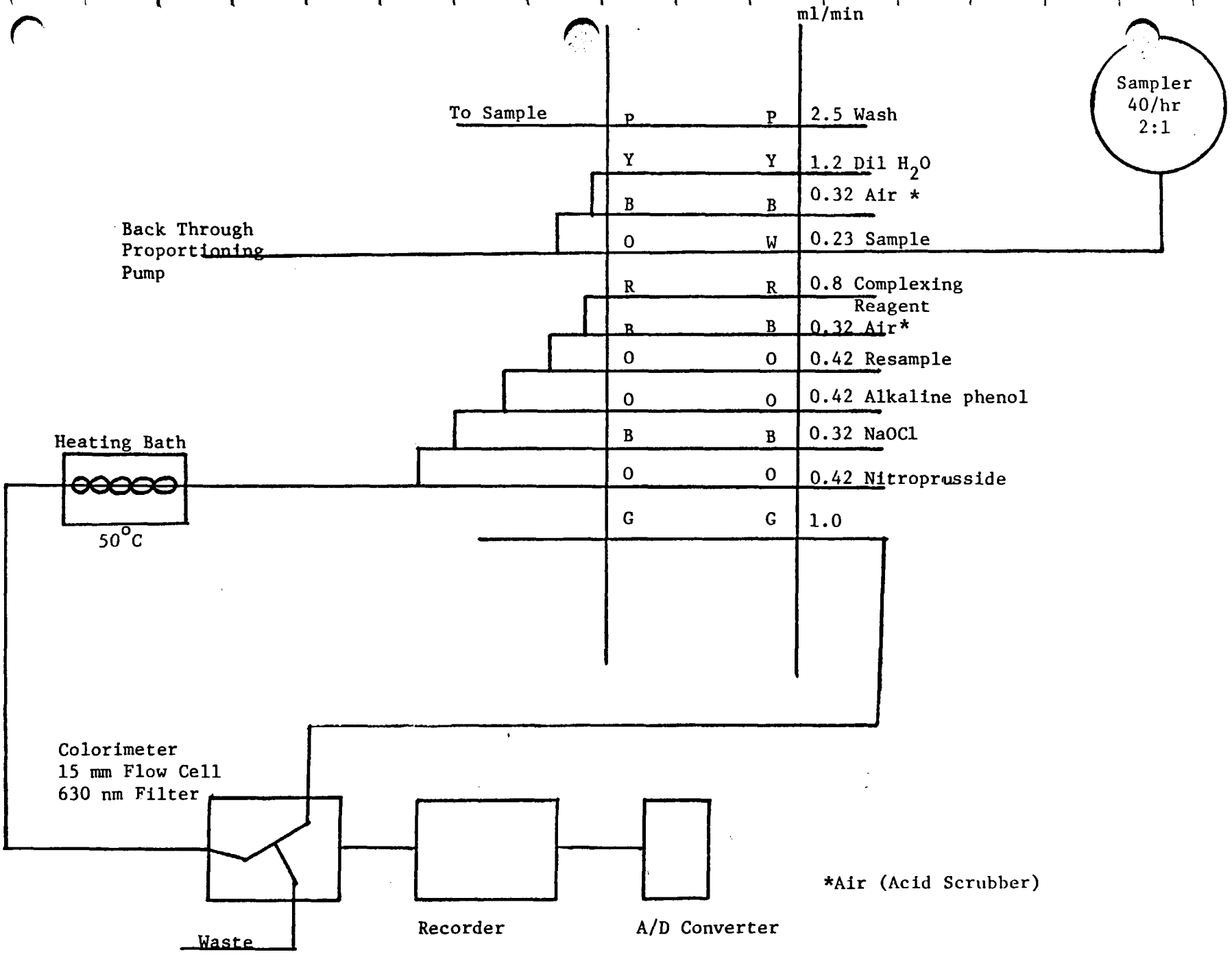
6. Booth, R. L., and Lobring, L. B., "Evaluation of the AutoAnalyzer II: A Progress Report" in Advances in Automated Analysis: 1972 Technicon International Congress, Vol. 8, p 7-10, Mediad Incorporated, Tarrytown, N.Y., (1973).

7. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 616, Method 604 (1975).

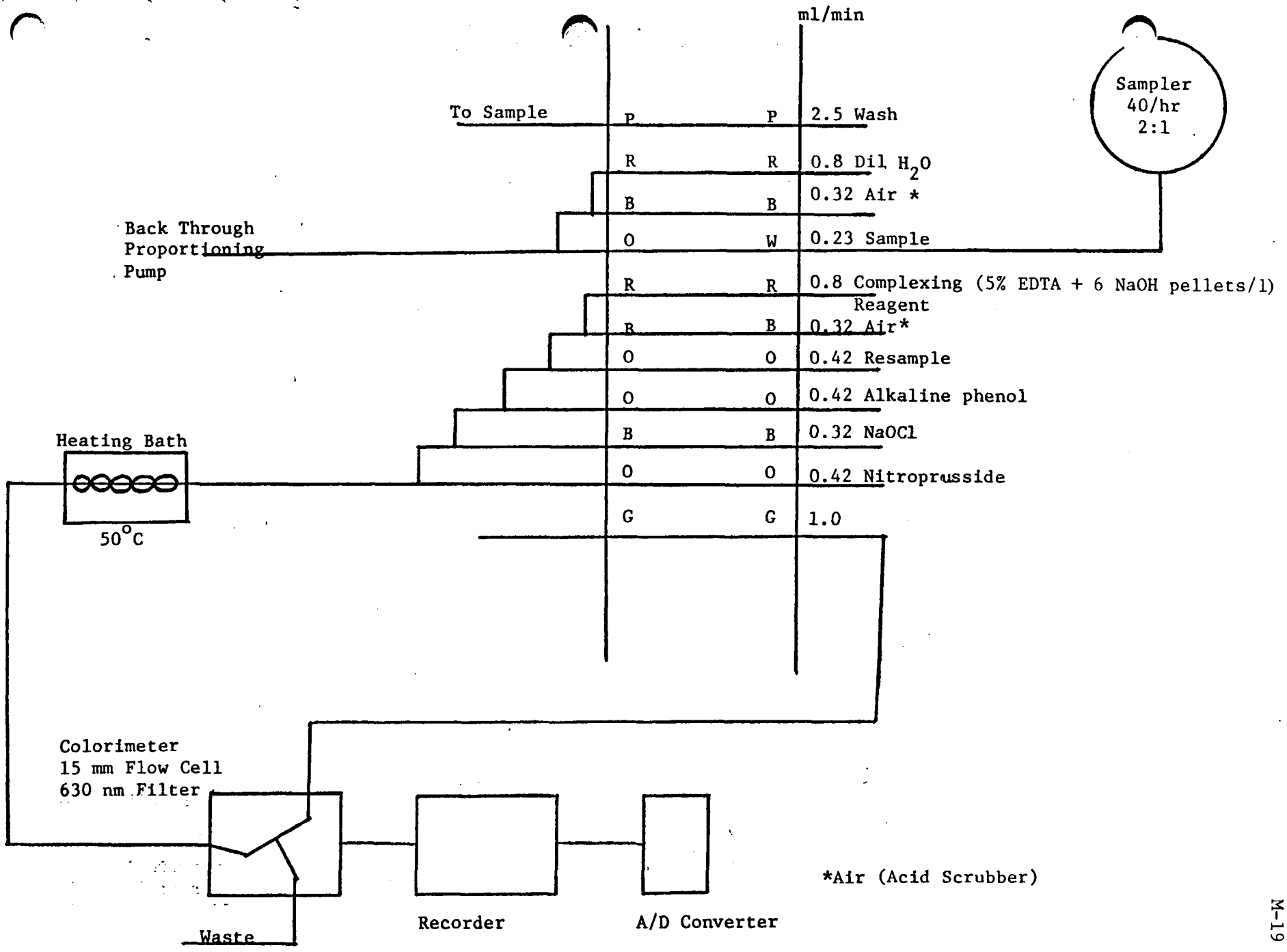


Nitrogen, Ammonia

Autoanalyzer II (Modified 11-15-85)



NITROGEN, AMMONIA
AUTOANALYZER II



NITROGEN, AMMONIA
AUTOANALYZER II (MODIFIED 12-16-81)

CHLORIDE
(Automated Ferricyanide Method)
(14th Ed. Std. Methods-ISBH Modifications)

ISBH Code No. C1-C-6-79
STORET No. 00940
Approved for NPDES

1. Scope of Application

   1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes. The applicable range is 1-100 mg/l Cl. Approximately 40 samples per hour can be analyzed.

2. Summary of Method

   2.1 Thiocyanate ion (SCN) is liberated from mercuric thiocyanate, through sequestration of mercury by chloride ion to form unionized mercuric chloride. In the presence of ferric ion, the liberated SCN forms highly colored ferric thiocyanate, in concentration proportional to the original chloride concentration.

3. Sample Handling and Preservation

   3.1 The samples are collected in one liter polyethylene bottles. No preservative is needed.

4. Comments

   4.1 No significant interferences.

5. Apparatus

   5.1 No significant change from referenced method.

6. Reagents

   6.1 Stock mercuric thiocyanate solution: Place 500 ml of methanol in a one liter volumetric flask. Add 4.17 g of mercuric thiocyanate, $Hg(SCN)_2$, and dissolve. Dilute to volume with methanol, mix, and filter through filter paper.

6.2 Stock ferric nitrate solution: Place 202 g of $Fe(NO_3)_3 \cdot 9H_2O$ in a one liter volumetric flask and add approximately 500 ml of distilled water. After dissolution, carefully add 22.2 ml of concentrated nitric acid to the flask and mix. Dilute to volume, mix, and filter through filter paper. Store in an amber reagent bottle.

6.3 Color reagent (prepare fresh daily): Place 75 ml of mercuric thiocyanate stock solution into a 500 ml volumetric flask. Add 75 ml of the stock ferric nitrate solution, dilute to volume with distilled water, and mix well.

6.4 Stock chloride solution: Place 0.8241 g NaCl dried at 140° C. in distilled water and dilute to one liter; 1 ml = 0.5 mg Cl.

6.5 Prepare a series of working standards by diluting suitable volumes of stock chloride solution to 500 ml with distilled water. The following dilutions are suggested:

ml of stock chloride solution	conc. mg Cl/l
10	10
20	20
40	40
60	60
80	80
100	100

6.6 Dilution water: Add Brij-35 to distilled water (5 drops per liter).

7. Procedure

7.1 No advance sample preparation is required. The manifold is set up as shown in Figure 1.

7.2 After the colorimeter and recorder warm up for approximately 30 minutes, establish a reagent baseline.

7.3 Place working standards in sampler in order of increasing concentrations. Complete filling of sampler tray with samples to be analyzed.

8. Calculations

8.1 the AutoAnalyzers are connected to a computer which receives the response signal from the colorimeter. After the type of curve fit is selected by the operator, the computer calculates the calibration curve by least squares method and generates concentration values for the samples, quality control solution, and laboratory blanks.

8.2 The response signal from the colorimeter is also connected to a strip chart recorder. The chart can be used to calculate concentration values by use of the overlay. The standard curve is prepared on the overlay by plotting the peak heights of standards against known concentrations. The concentration of the samples are obtained by comparing sample peak heights with the standard curve. The standard curve is not linear throughout the working range.

9. References

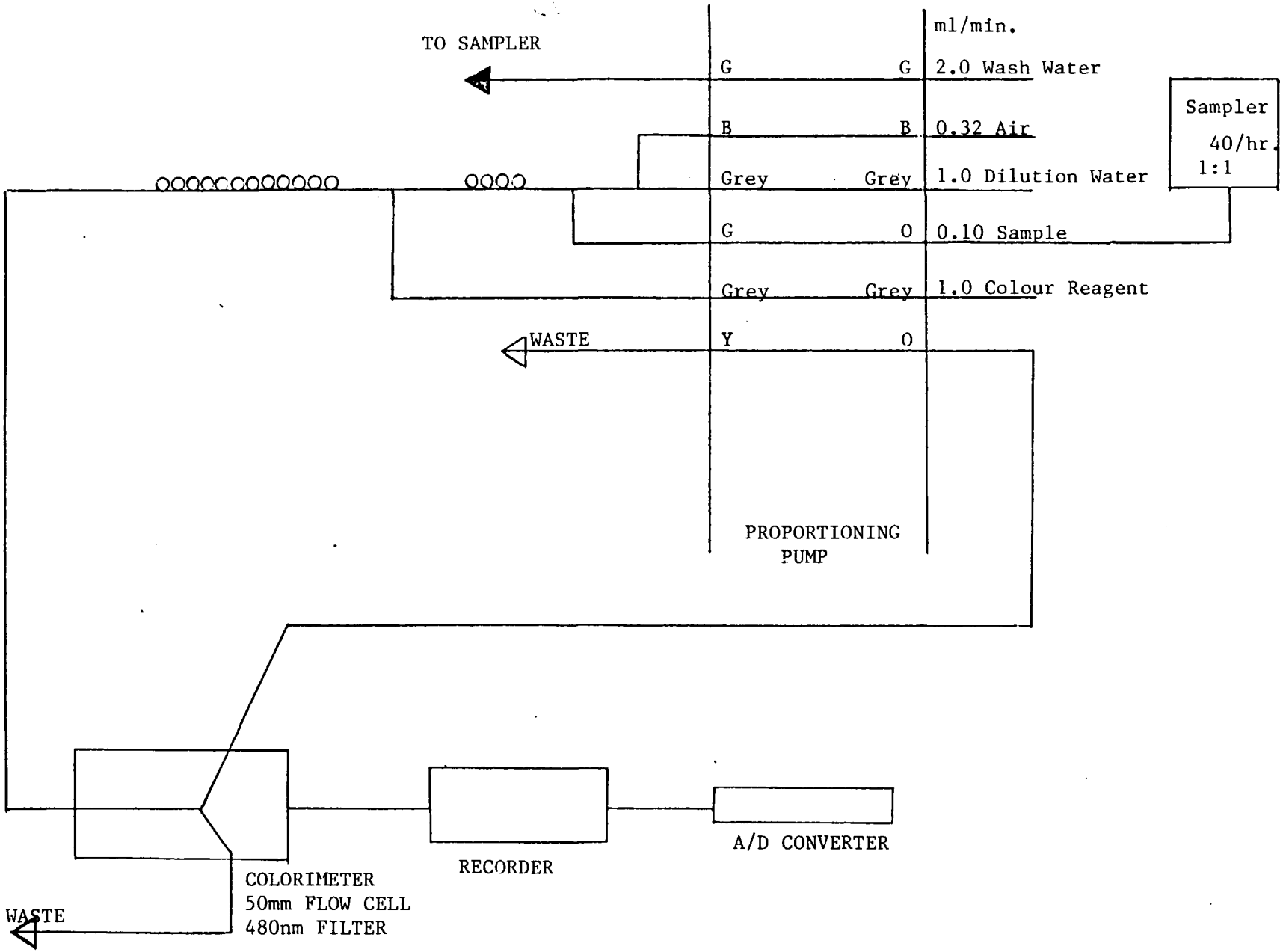
9.1 Standard Methods for the Examination of Water and Wastewater, 14th Edition, p. 613, Method 602, (1975)

9.2 Federal Register, Vol. 41, No. 232-Wednesday, December 1, 1976, p. 52781

9.3 "Methods for Chemical Analysis of Water and Wastes," 1974, p. 31, USEPA

9.4 J. E. O'Brian, "Automatic Analysis of Chlorides in Sewage," Waste Eng., 33, 670-672 (Dec. 1962)

lgb 7/26/82
W&S 2/Job I



CHLORIDE MANIFOLD
(FERRICYANIDE METHOD)
AUTOANALYZER II

(MODIFIED 11-19-85)

PHOSPHORUS, ALL FORMS
(Colorimetric, Automated, Ascorbic Acid)
ISBH Modification, 1979 EPA Manual

ISBH Code No. P-A-81
STORET NO. See Section 4
Approved for NPDES

1. Scope and Application

1.1 These methods cover the determination of specified forms of phosphorus in drinking, surface and saline waters, domestic and industrial wastes.

1.2 The methods are based on reactions that are specific for the orthophosphate ion. Thus, depending on the prescribed pre-treatment of the sample, the various forms of phosphorus given in Figure 1 may be determined. These forms are defined in Section 4.

1.2.1 Except for in-depth and detailed studies, the most commonly measured forms are phosphorus and dissolved phosphorus, and orthophosphate and dissolved orthophosphate. Hydrolyzable phosphorus is normally found only in sewage-type samples. Insoluble forms of phosphorus are determined by calculation.

1.3 The methods are usable in the 0.03 to 2.0 mg P/1 range. Approximately 40 samples per hour can be analyzed.

2. Summary of Method

2.1 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.

(Phosphorus, All Forms)

ISBH Code No. P-A-81
STORET NO. See Section 4
Approved for NPDES

2.2 Only orthophosphate forms a blue color in this test. Polyphosphates (and some organic phosphorus compounds) may be converted to the orthophosphate form by manual sulfuric acid hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by manual persulfate digestion. The developed color is measured automatically on the AutoAnalyzer.

3. Sample Handling and Preservation

3.1 If benthic deposits are present in the area being sampled, great care should be taken not to include these deposits.

3.2 Sample containers may be of plastic material; such as cubitainers, or of Pyrex glass.

3.3 If the analysis cannot be performed the same day of collection, the sample should be preserved by the addition of 2 ml 50% H_2SO_4 per liter and refrigeration at 4° C.

4. Definitions and Storet Numbers

4.1 Total Phosphorus (P)-all of the phosphorus present in the sample regardless of form, as measured by the persulfate digestion procedure (00665)

4.1.1 Total Orthophosphate (P-ortho)-inorganic phosphorus $\Sigma(PO_4)^{-3}\rfloor$ in the sample as measured by the direct colorimetric analysis procedure. (70507)

(Phosphorus, All Forms)

ISBH Code No. P-A-81
STORET NO. See Section 4
Approved for NPDES

4.1.2 Total Hydrolyzable Phosphorus (P-hydro)-phosphorus in the sample as measured by the sulfuric acid hydrolysis procedure, and minus predetermined orthophosphates. This hydrolyzable phosphorus includes polyphosphates $(P_2O_7)^{-4}$, $(P_3O_{10})^{-5}$, etc. plus some organic phosphorus. (00669)

4.1.3 Total Organic Phosphorus (P-org)-phosphorus (inorganic plus oxidizable organic) in the sample as measured by the persulfate digestion procedure, and minus hydrolyzable phosphorus and orthophosphate. (00670)

4.2 Dissolved Phosphorus (P-D)-all of the phosphorus present in the filtrate of a sample filtered through a phosphorus-free filter of 0.45 micron pore size and measured by the persulfate digestion procedure. (00666)

4.2.1 Dissolved Orthophosphate (P-D)-as measured by the direct colorimetric analysis procedure (00671)

4.2.2 Dissolved Hydrolyzable Phosphorus (P-D, hydro)-as measured by the sulfuric acid hydrolysis procedure and minus predetermined dissolved orthophosphates. (00672)

4.2.3 Dissolved Organic Phosphorus (P-D, org)-as measured by the persulfate digestion procedure, and minus dissolved hydrolyzable phosphorus and orthophosphate. (00673)

(Phosphorus, All Forms)

ISBH Code No. P-A-81
STORET NO. See Section 4
Approved for NPDES

4.3 The following forms, when sufficient amounts of phosphorus are present in the sample to warrant such consideration, may be calculated:

4.3.1 Insoluble Phosphorus (P-I)=(P)-(P-D). (00667)

4.3.1.1 Insoluble orthophosphate (P-I, ortho)=(P, ortho)-(P-D, ortho). (00674)

4.3.1.2 Insoluble Hydrolyzable Phosphorus (P-I, hydro)=(P, hydro)-(P-D, hydro). (00675)

4.3.1.3 Insoluble Organic Phosphorus (P-I, org)=(P, org)-(P-D, org). (00676)

4.4 All phosphorus forms shall be reported as P, mg/l, to the second place.

5. Interferences

5.1 No interference is caused by copper, iron, or silicate at concentrations many times greater than their reported concentration in sea water. However, high iron concentrations can cause precipitation of and subsequent loss of phosphorus.

5.2 The salt error for samples ranging from 5 to 20% salt content was found to be less than 1%.

5.3 Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than phosphorus. However, at concentrations found in sea water, it does not interfere.

5.4 Sample turbidity must be removed by filtration prior to analysis for orthophosphate. Samples for total or total hydrolyzable phosphorus should be filtered only after digestion. Sample color that absorbs in the photometric range used for analysis will also interfere.

6. Apparatus

6.1 Technicon AutoAnalyzer consisting of:

6.1.1 Sampler.

6.1.2 Analytical Cartridge (AAII).

6.1.3 Proportioning pump.

6.1.4 Heating bath, 37° C.

6.1.5 Colorimeter equipped with 15 or 50 mm tubular flow cell.

6.1.6 660 nm filter.

6.1.7 Recorder.

6.1.8 A/D Converter.

6.2 Autoclave.

6.3 Acid-washed glassware: All glassware used in the determination should be washed with 1:1 HCl and rinsed with distilled water. The acid-washed glassware should be filled with distilled water and treated with all the reagents to remove the last traces of phosphorus that might be absorbed on the glassware. Preferably, this glassware should be used only for the determination of phosphorus and after use it should be rinsed with distilled water and kept covered until needed again. If this is done, the treatment with 1:1 HCl and reagents is only required occasionally. Commercial detergent should never be used.

(Phosphorus, All Forms)

ISBH Code No. P-A-81
STORET NO. See Section 4
Approved for NPDES

7. Reagents

7.1 Sulfuric acid solution, 5N: Slowly add 70 ml of conc. H_2SO_4 to approximately 400 ml of distilled water. Cool to room temperature and dilute to 500 ml with distilled water.

7.2 Antimony potassium tartrate solution: Weigh 0.3 g $K(SbO)C_4H_4O_6 \cdot 1/2H_2O$, dissolve in 50 ml distilled water in 100 ml volumetric flask, dilute to volume. Store at 4° C in a dark, glass-stoppered.

7.3 Ammonium molybdate solution: Dissolve 4 g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 100 ml distilled water. Store in a plastic bottle at 4° C.

7.4 Ascorbic acid, 0.1M: Dissolve 1.8 g of ascorbic acid in 100 ml of distilled water. (Make fresh daily)

7.5 Combined reagent : Mix the above reagents in the following proportions for 100 ml of the mixed reagent: 50 ml of 5N H_2SO_4(7.1), 5 ml of antimony potassium tartrate solution (7.2), 15 ml of ammonium molybdate solution (7.3), and 30 ml of ascorbic acid solution (7.4). Mix after addition of each reagent. All reagents must reach room temperature before they are mixed and must be mixed in the order given. If turbidity forms in the combined reagent, shake and let stand for a few minutes until the turbidity disappears before processing. This volume is sufficient for 4 hours operation. Since the stability of this solution is limited, it must be freshly prepared for each run. NOTE 1: A stable solution can be prepared by not including the ascorbic acid in the combined reagent. If this is done, the mixed reagent (molybdate, tartrate, and acid) is pumped through the distilled water line and the ascorbic acid solution (30 ml of 7.4 diluted to 100 ml with distilled water) through the original mixed reagent line.

7.6 Sulfuric acid solution, 11 N: Slowly add 310 ml conc. H_2SO_4 to 600 ml distilled water. When cool, dilute to 1 liter.

7.7 Ammonium persulfate.

7.8 Acid wash water: Add 40 ml of sulfuric acid solution (7.6) to 1 liter of distilled water and dilute to 2 liters. (Not to be used when only orthophosphate is being determined.)

7.9 Phenolphthalein indicator solution (5 g/l): Dissolve 0.5 g of phenolphthalein in a solution of 50 ml of ethyl or isopropyl alcohol and 50 ml of distilled water.

7.10 Stock phosphorus solution: Dissolve 0.4393 g of pre-dried (150° C for 1 hour) KH_2PO_4 in distilled water and dilute to 1000 ml. 1 ml = 0.1 mg P.

7.11 Standard phosphorus solution: Dilute 50 ml of stock solution (7.10) to 1000 ml with distilled water. 1 ml = .005 mg P.

7.12 Prepare a series of standards by diluting suitable volumes of standard solutions to 200 ml with distilled water. The following dilutions are suggested:

ml of Standard Phosphorus Solution	mg P/l
20 ml of 0.3 ppm	0.03
20 ml of 0.5 ppm	0.05
20 ml of 1.0 ppm	0.1
40 ml of 1.5 ppm	0.3
20 ml of 5.0 ppm	0.5
40 ml of 5.0 ppm	1.0
60 ml of 5.0 ppm	1.5

7.13 Sodium chloride solution: Dissolve 20 g NaCl and 4 drops of Levor V in 1 liter of distilled water.

(Phosphorus, All Forms)

ISBH Code No. P-A-81
STORET NO. See Section 4
Approved for NPDES

8. Procedure

8.1 Phosphorus

8.1.1 Add 0.5 of sulfuric acid solution (7.6) to a 30 ml sample in a 25 x 150 mm culture tube.

8.1.2 Add 0.4 g of ammonium persulfate.

8.1.3 Heat for 20 minutes in an autoclave at 121° C (15-20 psi).

8.1.4 Determine phosphorus as outlined in (8.3.2) with acid wash water (7.8) in wash tubes.

8.2 Hydrolyzable Phosphorus

8.2.1 Add 0.5 of sulfuric acid solution (7.6) to a 30 ml sample in a 25 x 150 mm culture tube.

8.2.2 Heat for 30 minutes in an autoclave at 121° C (15-20 psi).

8.2.3 Cool and dilute the sample to 50 ml. If sample is not clear at this point, filter.

8.2.4 Determine phosphorus as outlined in (8.3.2) with acid wash water (7.8) in wash tubes.

8.3 Orthophosphate

8.3.1 Add 1 drop of phenolphthalein indicator solution (7.9) to approximately 50 ml of sample. If a red color develops, add sulfuric acid solution (7.6) drop-wise to just discharge the color. Acid samples must be neutralized with 1 N sodium hydroxide (40 g NaOH/1).

8.3.2 Set up manifold as shown in Figure 1 AAII.

8.3.3 Allow both colorimeter and recorder to warm up for 30 minutes. Obtain a stable baseline with all reagents, feeding distilled water through the sample line.

8.3.4 For the AAII system, use a 40/hr, 2:1 cam, and a common wash.

8.3.5 Place standards in Sampler in order of decreasing concentration. Complete filling of sampler tray with unknown samples.

8.3.6 Switch sample line from distilled water to Sampler and begin analysis.

9. Calculation

9.1 Prepare a standard curve by plotting peak heights of processed standards against known concentrations. Compute concentrations of samples by comparing sample peak heights with standard curve. Any sample whose computed value is less than 5% of its immediate predecessor must be rerun.

10. Data reduction is also done on computer support equipment.

Bibliography

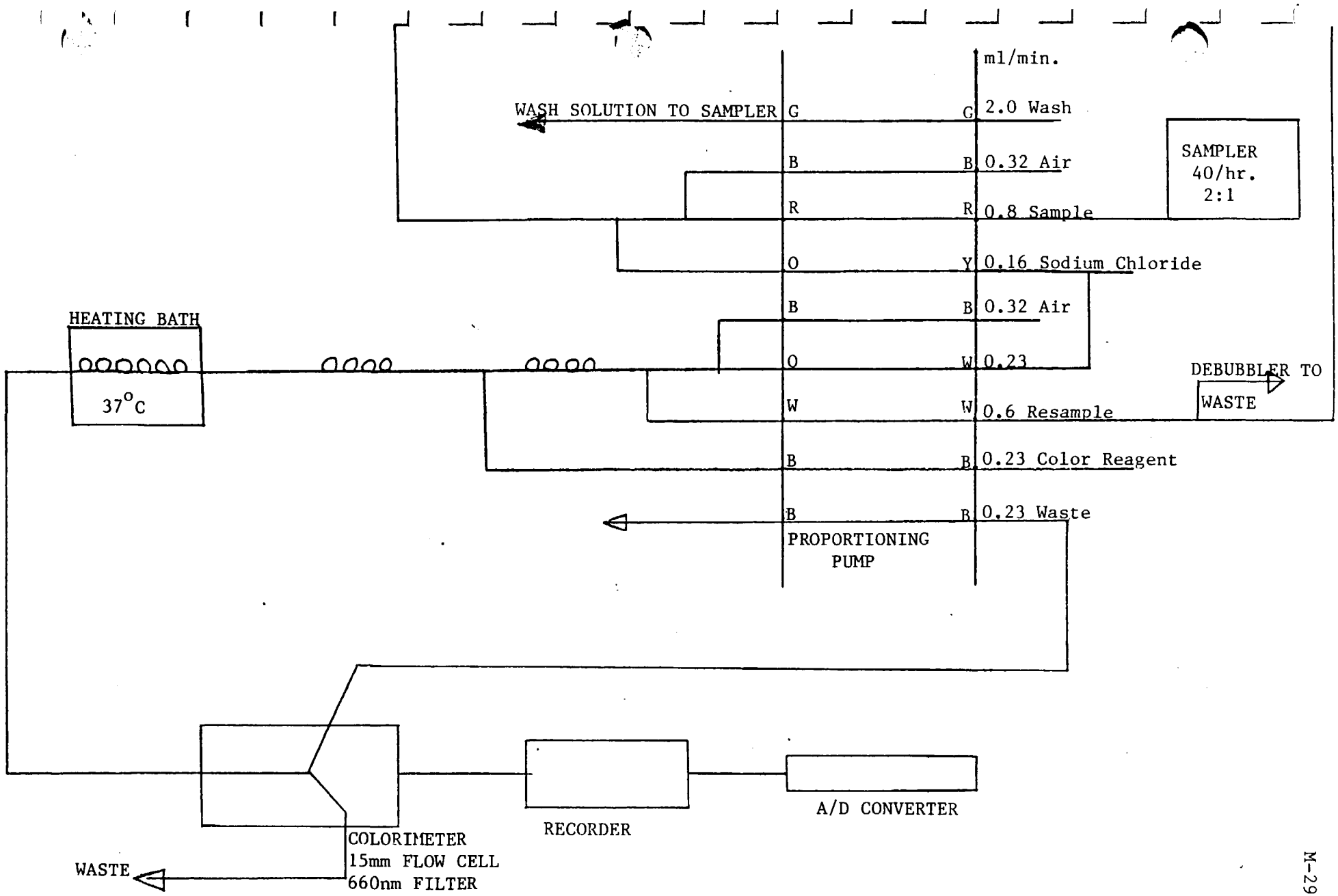
1. Murphy, J., and Riley, J., "A Modified Single Solution for the Determination of Phosphate in Natural Waters," Anal. Chem. Acta., 27, 31 (1962).

2. Gales, M., Jr., Julian, E., and Kroner, R., "Method for Quantitative Determination of Total Phosphorus in Water." Jour AWWA, 58, No. 10, 1363.

3. Lobring, L.B., and Booth, R.L., "Evaluation of the AutoAnalyzer II; A Progress Report," Technicon International Symposium, June 1972, New York, N.Y.

4. Annual book of ASTM Standards, Part 31, "Water," Standard D515-72, p. 388 (1976).

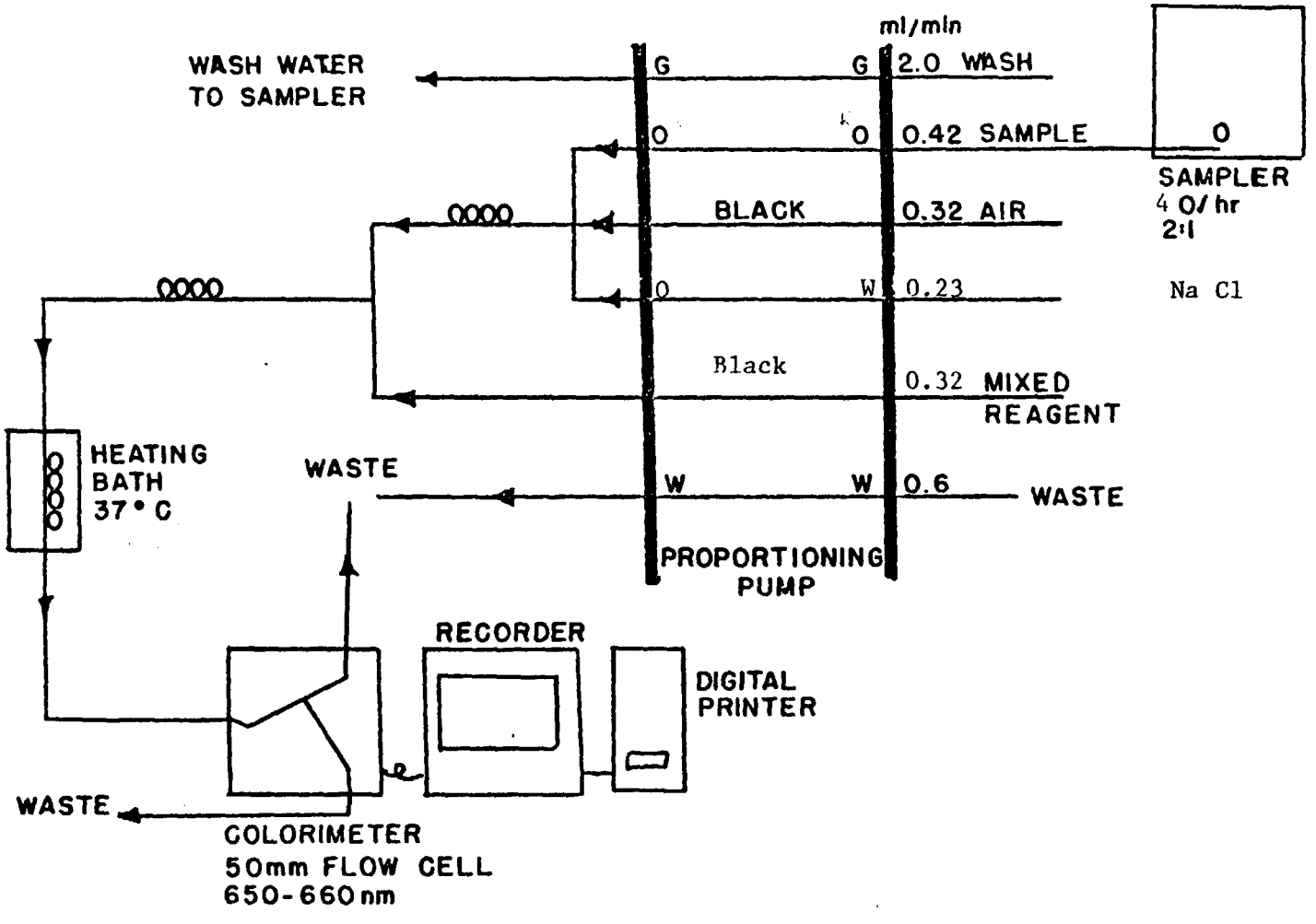
5. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p. 624, Method 606 (1975).



M-29

PHOSPHORUS MANIFOLD

AUTOANALYZER II (MODIFIED 11-19-85)



PHOSPHORUS MANIFOLD AA II

FIGURE I

NITROGEN, TOTAL KJELDAHL
(Ultramicro Semiautomated Method)

ISBH Code No. TKN-B-7-82
STORET NO. 00625

1. Scope and Application

1.1 This method is applicable to drinking water, surface water, domestic and industrial wastes.

1.2 The digested samples are analyzed by automated spectrophotometry at the rate of approximately 40 samples per hour.

1.3 The working range for the nitrogen is 0.1 to 10 mg/l, however, this range can be altered by modification of the digest volume or the manifold configuration.

2. Summary of Method

2.1 The manual digestion of the organic nitrogen is done in a Technicon block digestor. The method of choice is the use of fuming sulfuric acid in the presence of mercuric oxide catalyst to convert the organic nitrogen compounds to ammonia. The addition of potassium sulfate to the Kjeldahl method increases the digestion rate. The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert the nitrogenous compounds, of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semi-carbazones and some refractory tertiary amines.

2.2 The digested nitrogen compounds are analyzed for ammonia by a modification of the automated phenate method. In the phenate method, the indophenol blue reaction occurs as the ammonia reacts with the phenol and the hypochlorite to form a blue color. Sodium nitroprusside is used to intensify the color

3. Sample Handling and Preservation

3.1 Samples should be preserved with 2 ml of 50% H_2SO_4 per liter. Preserved samples should be analyzed as soon as possible.

3.2 Samples should be collected and stored in polyethylene bottles.

4. Interferences

4.1 Metals, such as mercury, complex ammonia and cause low results.

4.2 Substances, mostly metals, which are insoluble in basic solution can cause turbidity interference.

4.3 Metals such as manganese, which have two readily available oxidation states, catalyze the indophenol reaction and can enhance the color formation.

4.4 The addition of chelating agents such as citrate, EDTA, tartrate, and combinations of these, effectively decomplex the ammonia and complex the metals.

4.5 Nitroprusside has been found to stabilize the indophenol reaction and avoid sensitivity variations caused by metals.

5. Apparatus

5.1 Technicon BD-40 Block Digestor

5.2 Pyrex Test Tubes, Folin-Wu Digestion Tubes, 25 x 200 mm.

5.3 Vortex Genie Mixer

5.4 Technicon #114-0009-02 Rack (Modified)

5.5 Sampler IV

5.6 Analytical Cartridge (NH_3-N) AA II

5.7 Proportioning Pump III

5.8 Heating Bath, 40° C., AA I

5.9 Colorimeter, 15 mm Flow Cell, S10 Phototube, 630 nm Filters.

5.10 Recorder

5.11 Sulfuric Acid Trap (for air purification)

6. Reagents

All chemicals are ACS "Reagent" grade and all reagent water is deionized and distilled.

6.1 Digestion Solution: Dissolve 2 gm HgO in 25 ml of 6N H_2SO_4. Add 200 ml of conc. H_2SO_4 to 500 ml of the reagent water. While the strong acid solution is still hot, 134 gm of K_2SO_4 are dissolved in it and then the HgO solution is added. Cool the solution, bring to 1 liter with reagent water and store above 20° C. (No precipitation should occur)

6.3 Dilution Solution: Dilute 6.6 ml of 19N (50%) NaOH to 1 liter with reagent water.

6.4 (Replace sodium potassium tartrate solution with the following EDTA reagent.) Disodium ethylenediamine-tetraacetate (EDTA), (5%): Dissolve 50 g EDTA (disodium salt) and 20 ml 50% sodium hydroxide in 1 liter of distilled water.

6.5 Alkaline Phenol Solution: Dissolve 80 ml of phenol and 40 ml of 50% sodium hydroxide in 800 ml of reagent water, cool, and dilute to 1 liter. Store at 4° C.

6.6 Sodium Hypochlorite Solution: Dilute 125 ml of a bleach solution containing 5.25% NaOCl to 250 ml with distilled water. Prepare daily!

6.7 Sodium Nitroprusside Reagent: Dissolve 0.5 gm of sodium nitroprusside in 900 ml of reagent water and dilute to 1 liter. Store at 4° C.

6.8 Quality Control Sample: Solution of nicotinic acid of the desired strength.

6.9 Stock Ammonia Solution: Dissolve 3.819 gm of anhydrous ammonium chloride, dried at 105° C, in ammonia free water and dilute to 1 liter. (1 ml = 1 mg NH_3-N.)

6.10 Intermediate Standard: Dilute 100 ml of stock solution (6.9) to 1000 ml with ammonia free water. (1 ml = 0.01 mg NH_3-N.) Prepare daily.

6.11 Working Standards: Prepare daily.

ml of sol'n 6.10 per dig'n tube	mg/l NH_3-N
0.5	0.5
2.0	2.0
5.0	5.0
7.0	7.0
10.0	10.0

7. Procedure

7.1 Place 20 ml of preserved sample into the digestion tube (if the sample is nonhomogeneous, blend in a homogenizer before digestion) and place tube in the digestion rack.

7.2 Place 4-8 teflon boiling stones in each tube and 2 ml of digestion solution in each sample.

7.3 With each rack of samples, blanks (distilled deionized water), a series of standards, and two quality control samples should be included.

7.4 Place the rack of tubes in the block digestor and increase the time-temperature settings at the following rate:

7.4.1 Evaporate at a block temperature of 200° C for about 1 1/2 hour.

7.4.2 Increase temperature to 370° C and digest for about 2 1/2 hours.

7.5 Remove the rack of tubes, cool for at least 5 minutes, and add 20 ml of hot reagent water before the samples solidify. Mix samples on a vortex mixer.

7.6 The analytical cartridge and reagent tubes are set up according to the schematic. (Figure 1)

7.7 The colorimeter, recorder, and other equipment is warmed up for approximately 30 minutes with the reagents feeding through the lines.

7.8 A baseline is run with all reagents in place and the sampler wash solution feeding through the sample line.

7.9 The spans of the instrument are synchronized by using the maximum standard and the zero concentration.

7.10 The standards are arranged in the sample tray in increasing concentration and the unknown samples, which are digested, are then placed in the sampler tray. Also included in the tray are quality control samples, duplicates and blanks.

7.11 The sample line is switched to the sampler and the analytical run is started.

8. Calculations

8.1 The AutoAnalyzers are connected to a computer which receives the response signal from the colorimeter. After the type of curve fit is selected by the operator, the computer calculates the calibration curve by least squares method and generates concentration values for the samples, quality control solution, and laboratory blanks.

8.2 The response signal from the colorimeter is also connected to a strip chart recorder. The chart can be used to calculate concentration values by use of the overlay. The standard curve is prepared on the overlay by plotting the peak heights of standards against known concentrations. The concentrations of the samples are obtained by comparing sample peak heights with the standard curve. The standard curve is not linear throughout the working range.

9. Precision and Accuracy

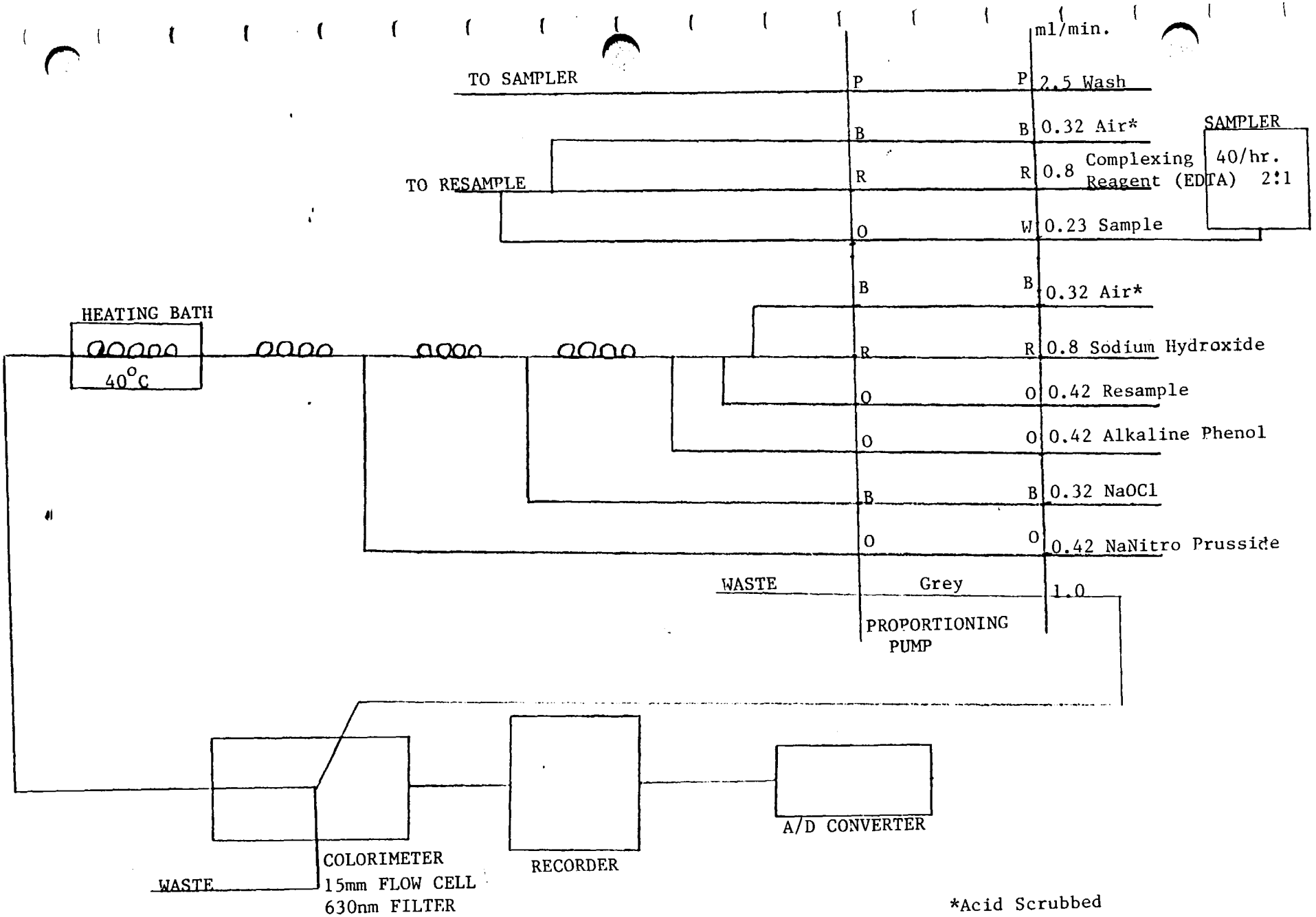
9.1 Detection Limit: This is defined as twice the standard deviation of the blank as determined by replicate blank analyses and the results of the blank for each run. Our detection limit at present is 0.1 mg/l N.

9.2 The precision and accuracy data for this analysis is obtained from the quality controls and real sample duplicates which are run 5-10% of the time.

9.3 Control limits are calculated at ±3 standard deviations from the mean value of the quality control standards.

10. References

10.1 "Ultramicro Semiautomated Method for Simultaneous Determination of Total Phosphorus and Total Kjeldahl Nitrogen in Wastewaters," Andrea M. Jirka, Mark Carter, Dorothy May, and Frederic Fuller, Environmental Science and Technology, p 1038, Vol. 10, No. 10, (October, 1976).

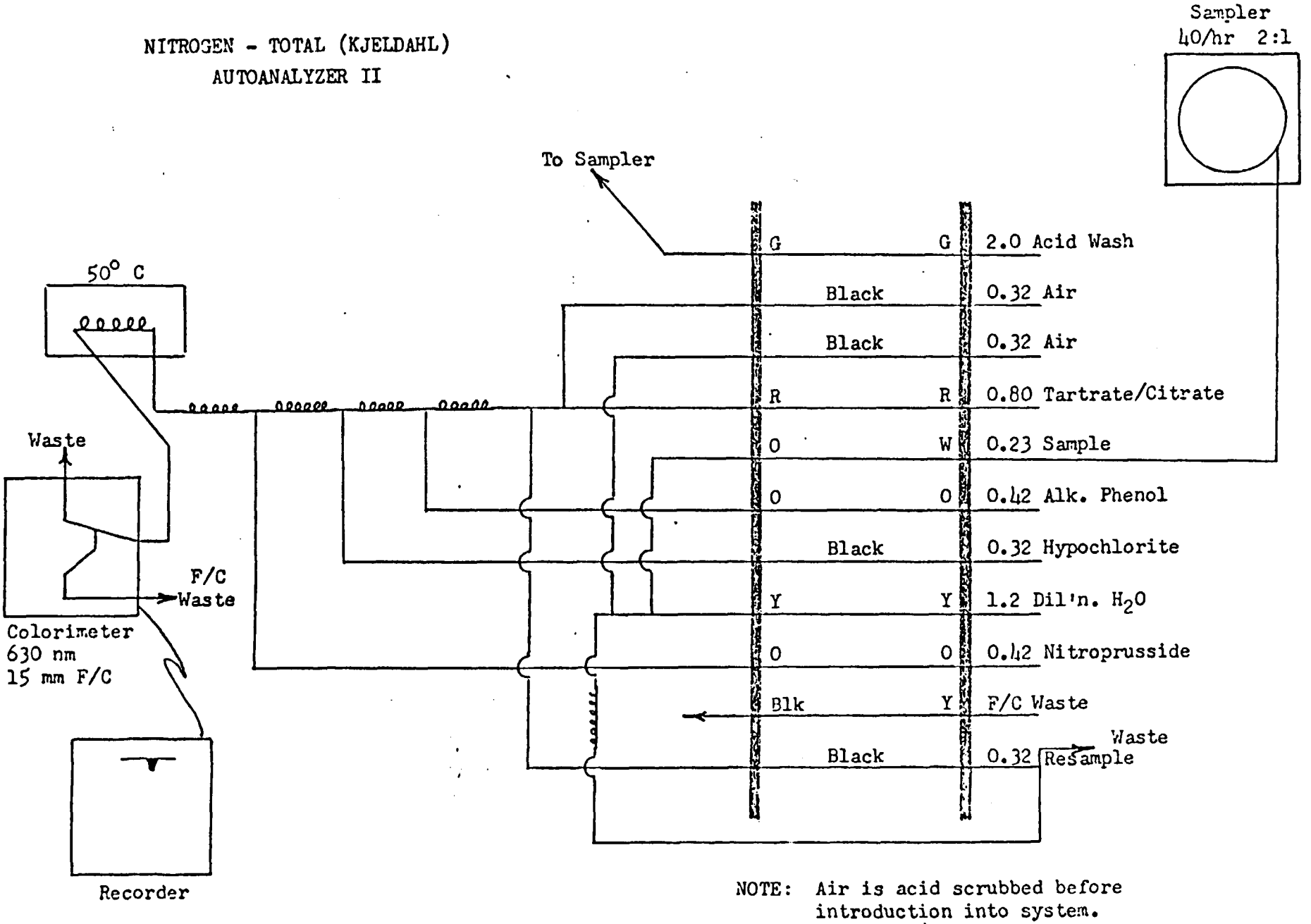


Nitrogen--Total (KJELDAML)

Autoanalyzer II (Modified 11-15-85)

FIGURE 1

NITROGEN - TOTAL (KJELDAHL)
AUTOANALYZER II



NOTE: Air is acid scrubbed before introduction into system.

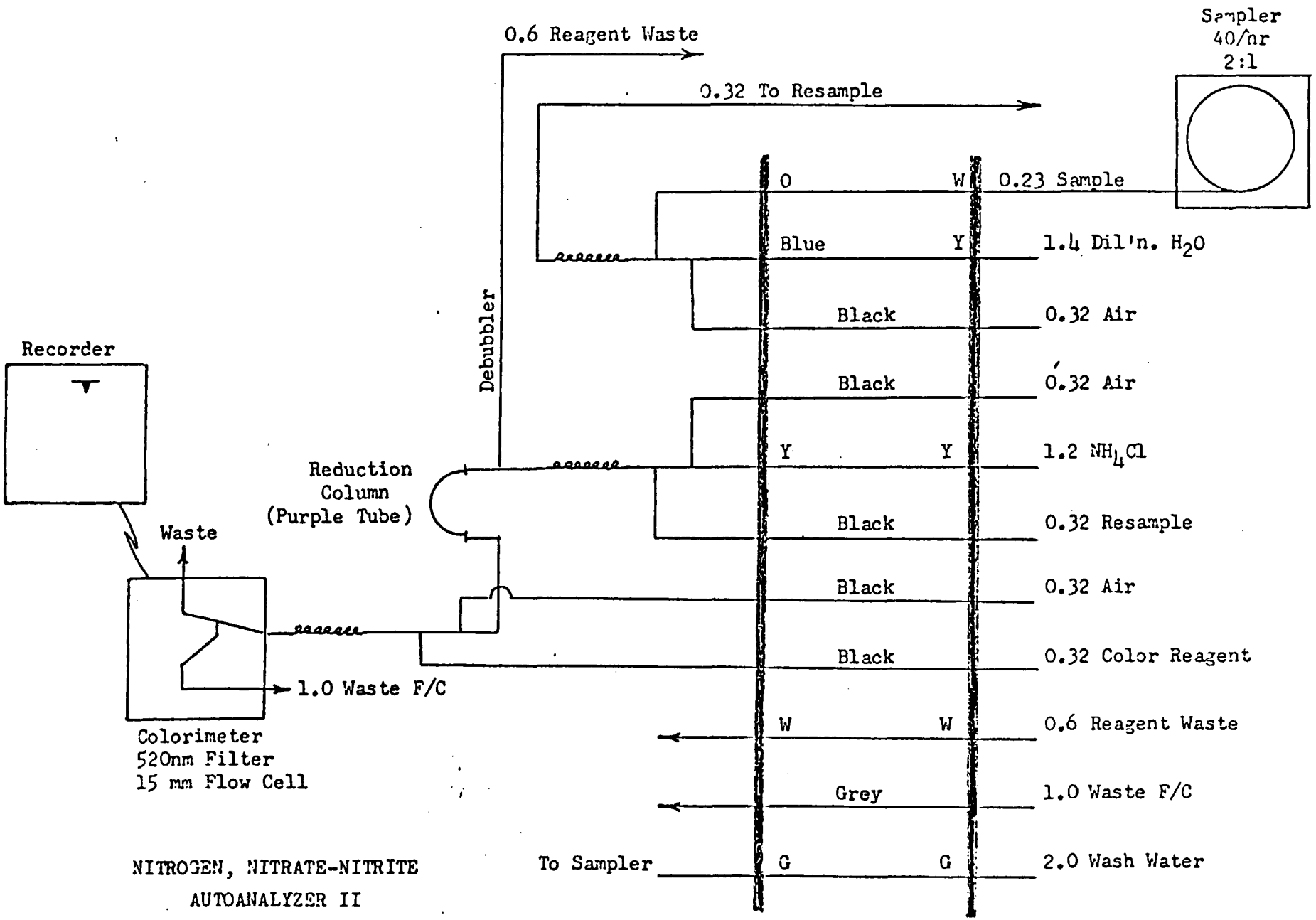


FIGURE 1

NITROGEN, NITRATE-NITRITE
AUTOANALYZER II

OIL AND GREASE
(Liquid-Liquid Extraction)
(Standard Methods, 13th Edition)

ISBH Code No. O,G-B-2-74
STORET No. 00556
Approved for NPDES

1. Scope and Application

1.1 This method includes the measurement of extractable matter, usually oil and grease in solution or suspension, from surface waters, industrial wastes, and sewages.

2. Summary of Method

2.1 The sample is acidified to a low pH (less than 3) with sulfuric acid and extracted with trichlorotrifluoroethane (Freon 113) by a liquid-liquid extraction in a separatory funnel. The solvent is evaporated from the extract and the residue is weighed.

3. Comments and Interferences

3.1 Low boiling fractions or the light hydrocarbons are lost in this method of analysis for oil and grease. Some lubricating oil fractions evaporate at the temperature necessary for removal of the extraction solvent. Kerosene is still more volatile and gasoline cannot be determined by the organic extraction method.

3.2 Some oils in natural waters may be derived from the decomposition of plankton and higher forms of aquatic life.

3.3 Most heavy oils and greases are insoluble in water but may be emulsified or saponified by detergents, alkalis or other chemicals.

3.4 Solvents are not selective in dissolving only oil and greases; and they can vary considerably in their ability to dissolve not only oil and grease, but other organic substances as well.

3.5 On standing, solvents tend to form oxidation products which leave a gummy residue on evaporation.

3.6 When an emulsion of saponified oil or grease is present in the sample, acidification to pH 1 and saturation with sodium chloride aids in breaking up this emulsion.

4. Sample Handling and Preservation

4.1 A clean 500 ml wide mouth glass bottle should be filled with sample and acidified with 2 ml of 50% sulfuric acid to inhibit bacterial activity. The complete sample should be used for the oil and grease analysis.

5. Apparatus

5.1 Separatory funnel, 1 liter, with teflon stopcock

5.2 Steam bath or electric heating mantle

5.3 Evaporating dish, Coors #2, porcelain

5.4 Filter paper, phase separating, Whatman PS-1

5.5 125 ml glass bottles.

6. Reagents

6.1 Sulfuric acid, 18 N

6.2 Trichlorotrifluoroethane (Freon 113)

7. Procedure

7.1 Measure total sample in the 1 liter graduated cylinder and pour into the 1 liter separatory funnel.

7.2 Rinse the graduated cylinder with 15 ml Freon, pour into the glass collection bottle, and after rinsing, add the washings to the separatory funnel.

7.3 Add an additional 15 ml of Freon to the separatory funnel and shake vigorously for 2 minutes. Allow the organic layer to separate.

7.4 Filter the solvent layer into a clean 125 ml bottle.

NOTE: The phase separating filter paper is placed in the funnel, prewashed with a portion of solvent, and the solvent rinse is discarded.

7.5 Again add 30 ml of Freon to the separatory funnel and agitate for 2 minutes. Allow the solvent layer to separate.

7.6 Filter the Freon extract into the 125 ml bottle using the same filter paper. Add 30 ml of Freon to the separatory funnel and agitate for 2 minutes. Allow the solvent layer to separate and filter into the 125 ml bottle. Rinse the filter paper with 10 ml of the solvent.

7.7 Add the combined Freon extracts to a tared evaporating dish and place the dish on a steam plate or covered steam bath which has been modified to produce a temperature of 70-80° C.

7.8 After the solvent is evaporated, place the dish in a dessicator for 30 minutes and weigh.

NOTE: A blank should be run on the Freon to compensate for any solvent residue. A volume of Freon, equivalent to the amount used in the sample extraction and washings, should be evaporated in a tared dish. The remaining residue should be weighed and this result taken into consideration in the calculation of the oil and grease in the sample.

8. Calculation

$$\text{mg/l Oil and Grease} = \frac{(A-B) \times 1000}{\text{ml sample}}$$

A = weight of sample residue

B = weight of solvent residue

9. References

9.1 Federal Register, Vol. 38, No. 199, (October 16, 1973), Part II, Environmental Protection Agency, Water Programs.

9.2 "Standard Methods for Examination of Water and Wastewater," 13th Edition, pp 254-256, Method 137.

SULFATE
(Methylthymol Blue Automated Method)
(14th Edition "Standard Methods")

ISBH Code No. B-11-81
STORET No. 00945
Approved for NPDES

1. Scope and Application

   1.1 This method is applicable to potable, surface, and saline waters as well as domestic and industrial wastes.

   1.2 The method is suitable for a range of 1 - 100 mg/l SO_4. This range can be modified by making changes in the sulfate manifold. Approximately 30 samples per hour can be analyzed.

2. Summary of Method

   2.1 In this method for determining sulfate, it is necessary to remove interference by passing the sample through a cation-exchange column. The sample containing sulfate is then reacted with barium chloride to form barium sulfate at a pH of 2.5 to 3.0. Excess barium reacts with methylthymol blue to form a blue-colored chelate at a pH of 12.5 to 13.0. The uncomplexed methylthymol blue is gray in color, and when it is chelated with barium it forms a blue color. Initially the barium chloride and methylthymol blue are present in equimolar amounts. Therefore, the amount of uncomplexed methylthymol blue is equal to the sulfate present.

3. Sampling and Handling

   3.1 No preservative is needed.

   3.2 Samples are collected in polyethylene bottles.

   3.3 Samples should be stored at low temperature (4° C.).

4. Interference

4.1 Color, turbidity, cations such as calcium, aluminum, and iron interfere, but are removed by the cation-exchange column.

5. Apparatus

5.1 Technicon AutoAnalyzer consisting of:

5.1.1 Sampler I with 40/hr - 1:1 cam.

5.1.2 Sulfate Manifold.

5.1.3 Proportioning Pump.

5.1.4 Colorimeter equipped with 50 mm tubular flow-cell and 460 nm filters.

5.1.5 Recorder.

5.1.6 A/D Converter.

6. Reagents

6.1 Barium Chloride Solution: Dissolve 1.526 gm $BaCl_2.2H_2O$ in 900 ml of distilled water and dilute to 1 liter. Store in a polyethylene bottle.

6.2 Hydrochloric Acid, 1.0 N Solution: Add 83 ml of conc. HCl to 800 ml of distilled water and dilute to 1 liter.

6.3 Methylthymol Blue solution: Dissolve 0.1182 gm of methylthymol blue in 25 ml of barium chloride solution (6.1). Add 4 ml of 1.0 N HCl solution (6.2), which produces a bright orange color. Add 71 ml of distilled water and dilute to 500 ml with ethanol (95% reagent grade). The pH of this solution should be 2.6. Store in a brown glass bottle in the refrigerator and prepare weekly.

6.4 Buffered EDTA Solution: Dissolve 6.75 gm NH_4Cl in 500 ml of distilled water. Add 75 ml of conc. NH_4OH and dilute to 1 liter with distilled water. Add and dissolve 40 gm of tetrasodium EDTA. Store in a polyethylene bottle.

6.5 Sodium Hydroxide, 0.18 N Solution: Add 12 ml of 50% NaOH to 800 ml of distilled water and dilute to 1 liter. Prepare fresh weekly.

6.6 Stock Sulfate Solution, 100 mg/l: Dissolve 1.479 gm of anhydrous Na_2SO_4 in 500 ml of distilled water and dilute to 1 liter.

6.6.1 Prepare a series of working standards by diluting volumes of stock solution to 200 ml with distilled water. The following dilutions are suggested:

ml Stock Sol'n	mg/l SO_4
4.0	20.
8.0	40.
12.0	60.
16.0	80.
20.0	100.

6.7 Ion-exchange Column: The column is made of a length of glass tubing 7.5 inches long x 2.0 mm ID x 3.6 mm OD. Wash the cation-exchange resin three times with distilled water to remove the fines. Next fill the column with the resin, being careful not to allow air to become trapped in the column. Place glass wool plugs in each end to prevent resin from escaping. Use Bio Rex 70, 20-50 mesh, Na+ form.

6.8 Dilution Water: Distilled water.

7. Procedure

7.1 No advance sample preparation is required. Set up the manifold as shown in the schematic. (Figure 1)

7.2 Allow the colorimeter and recorder to warm up for 30 minutes.

7.3 Run a baseline with all reagents, feeding distilled water through the sample line, then place the cation-exchange column in place. Adjust the colorimeter to obtain a stable baseline and set the span on the recorder to obtain the working range.

7.4 Sample at the rate indicated on the schematic.

7.5 Place the working standards in the sampler tray in increasing order of sulfate concentration. Complete filling the sampler tray with unknown samples.

7.6 Run at least two quality control samples and two duplicate samples in each tray.

7.7 Start the sample run once a stable baseline is obtained.

7.8 At the end of the sample run the system should be purged with a solution of buffered EDTA. This can be done by placing the methylthymol blue line and the NaOH line in water for a few minutes and then into the EDTA for ten minutes. Then wash with water for fifteen minutes before shutting down. Remove the resin column while full of water if it is to be used again. Rinsing also with 1.0 N HCl in the same manner as EDTA aids in removal of build-up in the flow-cell.

8. Calculations

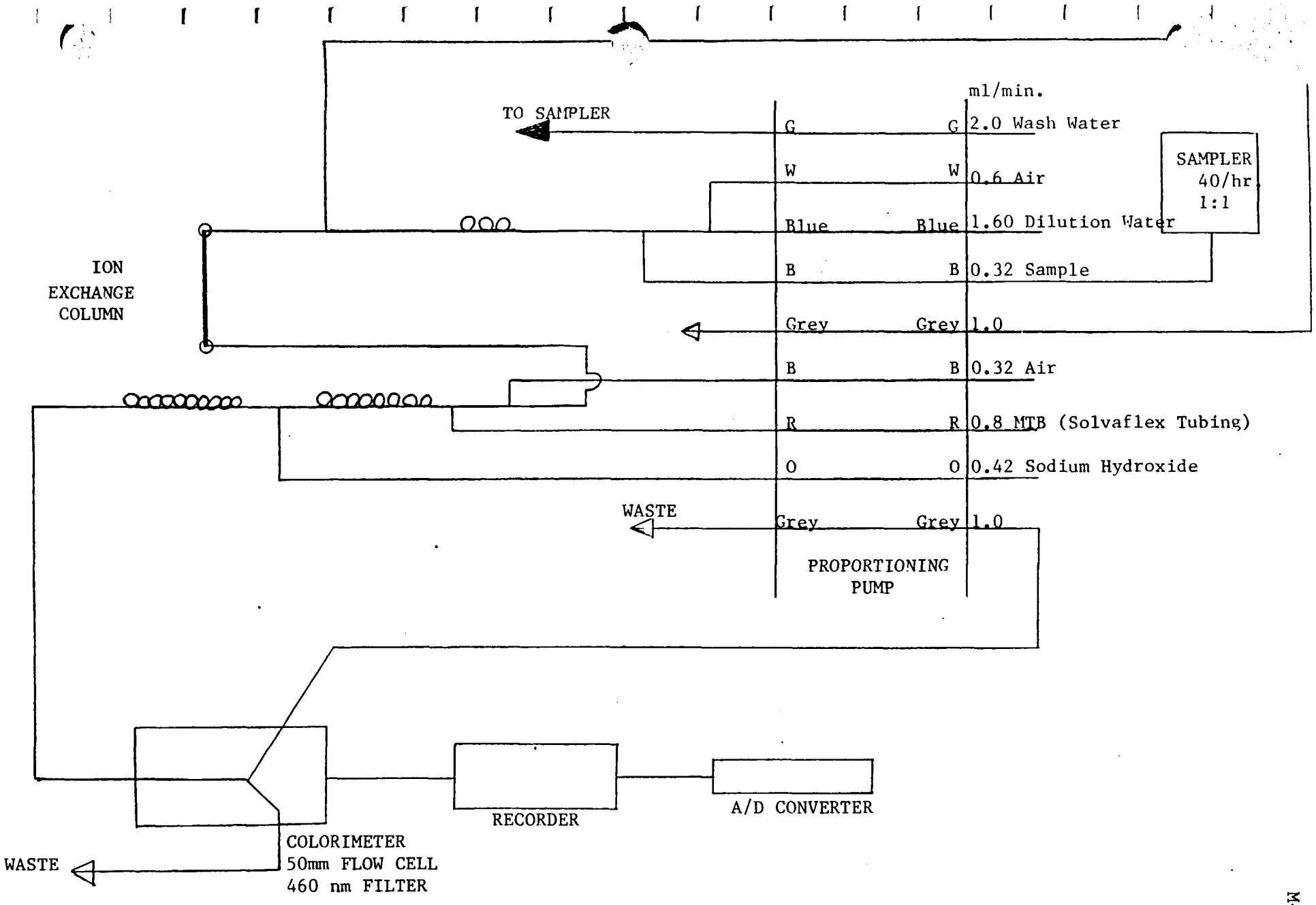
8.1 The AutoAnalyzers are connected to a computer which receives the response signal from the colorimeter. After the type of curve fit is selected by the operator, the computer calculates the calibration curve by least squares method and generates concentration values for the samples, quality control solution, and laboratory blanks.

8.2 The response signal from the colorimeter is also connected to a strip chart recorder. The chart can be used to calculate concentration values by use of the overlay. The standard curve is prepared on the overlay by plotting the peak heights of standards against known concentrations. The concentration of the samples are obtained by comparing sample peak heights with the standard curve. The standard curve is not linear throughout the working range.

9. References

9.1 "Standard Methods for the Examination of Water and Wastewater," 14th Edition, p 628, Method 607 (1975).

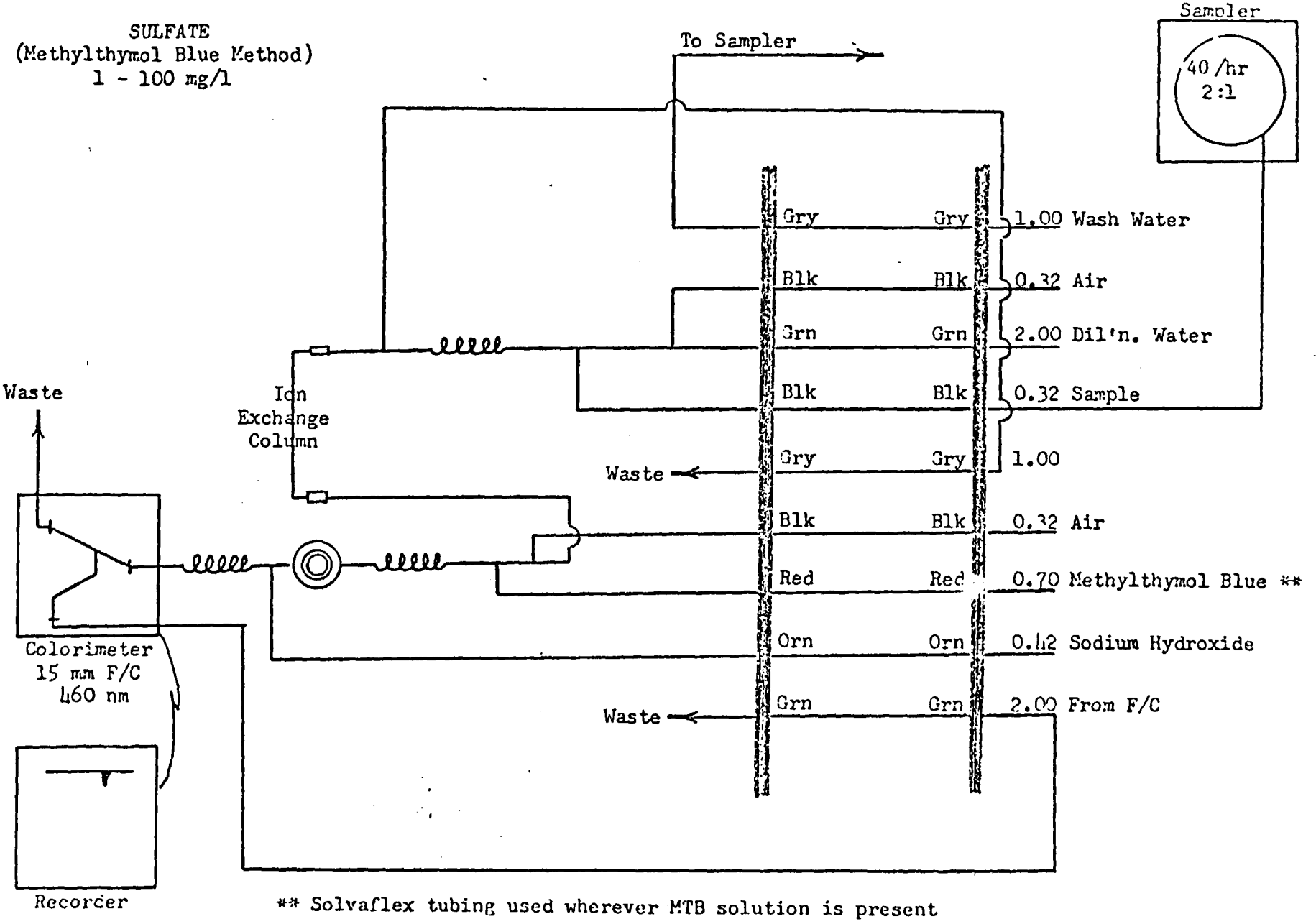
9.2 Technicon Industrial Systems. "Sulfate in Water and Wastes," (Industrial Method AA II 118-71W), December 1972.



SULFATE MANIFOLD
(METHYL THYMOL BLUE METHOD)

AUTOANALYZER II (MODIFIED 11-19-85)

SULFATE
(Methylthymol Blue Method)
1 - 100 mg/l



** Solvaflex tubing used wherever MTB solution is present

Figure 1

Appendix E
FIELD MEASUREMENT OF pH

FIELD MEASUREMENT OF pH

Method: Electrometric

Reference: EPA 1979, Page 150.1

Sensitivity: 0.1 pH unit

Optimum Range: 1-12 pH units

Sample Handling: Determine on-site or within 6 hours.

Reagents and Apparatus:

1. pH meter (Orion Model 211 Mini pH meter).
2. Combination electrodes
3. Beakers or plastic cups.
4. pH buffer solutions, pH 4, 7, and 10.
5. Deionized water in squirt bottle.
6. All glassware soap and water washed, followed by two hot water rinses and two deionized water rinses.

Calibration:

1. Place electrode in pH7 buffer solution.
2. After allowing several minutes for meter to stabilize, turn calibration dial until a reading of 7.00 is obtained.
3. Rinse electrode with deionized water and place in pH4 or pH10 buffer solution.
4. Wait several minutes and then turn slope adjustment dial until a reading of 4.00 or 10.00 is obtained.
5. Rinse electrode with deionized water and place in pH7 buffer. If meter reading is not 7.00, follow Steps 2-5 again.

Procedure:

1. Calibrate meter using calibration procedure.
2. Pour the sample into a clean beaker or plastic cup.

3. Rinse electrode with deionized water between samples. Recheck calibration with pH7 buffer solution after every 5 samples.

4. Immerse electrode in solution. Make sure the white KCl junction on side of electrode is in the solution. The level of electrode solution should be one inch above sample to be measured.

Notes:

1. When calibrating the meter, use pH buffers 7 and 4 for samples with pH < 8, and buffers 7 and 10 for samples with pH > 8. If meter will not read pH4 or 10, something may be wrong with the electrode. Return it to the lab with a note.

2. pH is a temperature dependent analysis. Therefore, temperatures of buffers and samples should be within about 2°C. For refrigerated or cool samples, use refrigerated buffers to calibrate meter.

3. Weak organic and inorganic salts and oil and grease are interferences in pH measurements. If oil and grease are visible, note on data sheet. Clean electrode with soap and water, followed by 10% HCl. Then recalibrate meter.

4. When not in use, the electrode should be stored in pH4 buffer.

5. Before going into the field:

   a) Check batteries;
   b) Do a quick calibration at pH7 and 4 to check electrode;
   c) Obtain fresh solutions.

6. Following field measurements:

   a) Report any problems;
   b) Compare with previous data;
   c) Clean all dirt off of meter and inside case;
   d) Make sure electrode is stored in pH4 buffer.

ATTACHMENT 1

INSTRUCTION MANUAL
ORION MODEL 211
pH METER

INSTRUCTION MANUAL

model 211
digital pH meter

ORION RESEARCH

contents

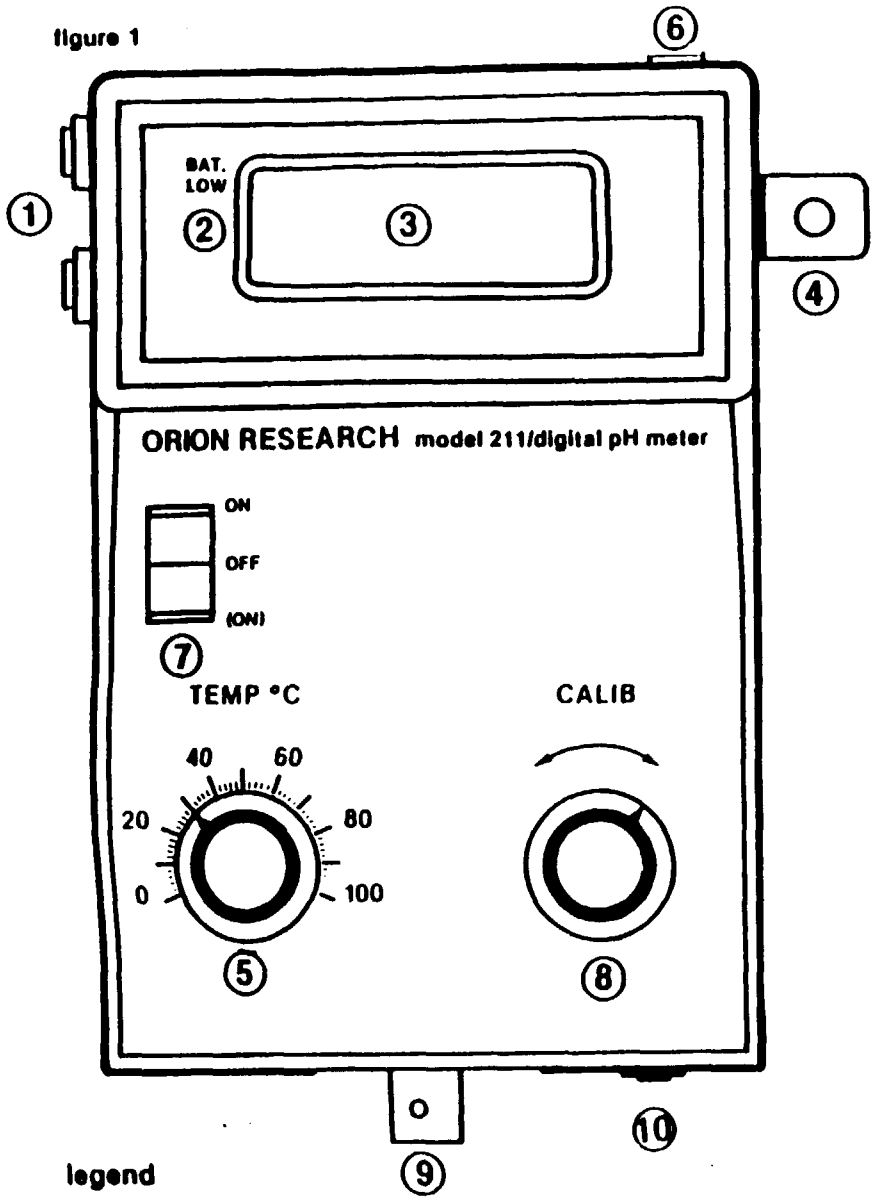
repair/service

For information on repair or replacement of this instrument, contact Orion Research toll-free. Ask for Customer Service.

ORION RESEARCH INCORPORATED
Customer Service
840 Memorial Drive
Cambridge, Massachusetts 02139 U.S.A.
800-225-1480 (Continental U.S.)
617-864-5400 (Massachusetts, Alaska, Hawaii, Canada)
Telex: 921466

Form IM211/3860
Printed in U S A

figure 1



legend

1. strip chart recorder binding posts
2. BAT LOW
3. LC display
4. support rod clip
5. temperature indicator control
6. AC line adapter input
7. function control
8. calibration control
9. electrode connector
10. slope control

introduction

The Model 211 is a battery- or line-operated (110/220 V AC adapter) digital pH meter for field or laboratory use. The meter is complete with strip chart recorder binding posts and is supplied with an unbreakable, gel-filled combination pH electrode, one packet of pH 7 buffer powder, one bottle for pH 7 buffer, one bottle for distilled water, support rod, electrode holder, AC adapter, six 1.5 V batteries, shorting plug, and carrying case.

instrument description

See figure 1.

1. **strip chart recorder binding posts:** black post is low (ground) and red post is high input side of recorder. See page 8.
2. **BAT LOW:** an arrow pointing towards BAT LOW appears on the display when battery requires replacement.
3. **LC display:** pH display over the range of 0 - 14 with ± .01 pH units resolution
4. **support rod clip:** holds steel rod used to mount electrode holder.
5. **temperature indicator control (TEMP °C):** compensates for variation in electrode slope or temperature changes. Used in two-buffer calibration.
6. **AC line adapter input:** jack used to insert AC line adapter. With AC line adapter operational, the internal battery is bypassed.
7. **function control:** rocker switch with three positions - ON, OFF and (ON) Depress (ON) for a momentary reading. The switch will return to OFF when released.
8. **calibration control (CALIB):** used to calibrate the meter with buffers of known pH.
9. **electrode connector:** accepts BNC connector from pH electrode.
10. **slope control:** screwdriver adjustment used to set second buffer in two-buffer calibration.

instrument set-up

support rod

1. Insert steel support rod into the hole in the support rod clip on side of the meter.
2. Mount electrode holder on the rod by pinching to compress the spring. Release to hold in place.

power source

The Model 211 operates on six nonrechargeable 1.5 volt batteries or on 110 or 220 ± 20% V with an approved AC adapter (specify voltage when ordering). Low battery is indicated by the BAT LOW indicator on the display.

NOTE: Batteries are not rechargeable - use of line adapter whenever possible will prevent the unit's batteries from being discharged. If battery operation is desired, follow installation instructions under **battery replacement**.

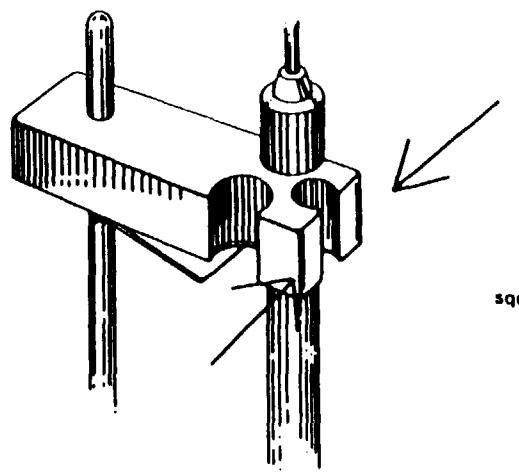
meter check-out

1. Install six AA batteries in the meter. Orient the (+) and (-) battery terminals to match the orientation shown in the battery compartment.
2. Depress ON button on the front panel. If the BAT. LOW indicator on the front display lights up, the batteries must be replaced.
3. If battery mode is not to be used, disregard steps 1 and 2. Insert pin end of appropriate AC line adapter into the meter, and the other end into the appropriate grounded AC line receptacle.
4. Attach BNC shorting plug to BNC input on the bottom side of the meter. Depress ON button on the front panel. Turn CALIB knob so display reads a steady 7.00. If this cannot be done consult ORION Technical Service.
5. Remove the shorting plug. Successful completion of steps 1-4 show the [illegible] is ready for use.

connecting electrode

1. Insert the BNC connector into the electrode jack on the bottom panel of the meter. Turn connector clockwise until it seats firmly.
2. Mount electrode in the electrode holder by spreading the electrode clip open and sliding the electrode into the holder so that the clip closes on electrode cap. See figure 2.
3. Follow **measurement procedures** to use the meter to measure pH.
4. Disconnnect electrode by turning connector counterclockwise until released from pin.

figure 2



squeeze as shown to insert electrode

measurement procedures

general measurement technique

temperature: All samples and buffers should be at the same temperature, as small variations in temperature can cause errors in measurement. The slope of the pH electrode, the potential of the reference electrode, and the pH of the buffer are temperature-dependent.

cleaning electrodes: Electrode should be rinsed and shaken between measurements to remove drops and to prevent solution carryover.

stirring: Stir measured solutions moderately to obtain good contact between the glass bulb and the solution. Insert electrode to a depth of about 3 cm.

pH measurements

single-buffer standardization
(where maximum precision is not required)

NOTE: For maximum accuracy it is recommended that a two-buffer calibration be performed once at the beginning of each day (see page 7). This procedure ensures the correct setting of the slope control. Subsequent measurements during the day may be made using a single point calibration.

1. Place the electrode in a buffer solution whose pH is near the expected pH of the sample. Insert electrode to a depth of about 3 cm and stir moderately.
2. Set the **temperature indicator control** to the temperature of the buffer.
3. Set the **function control** to **ON** and allow the buffer reading to stabilize. Adjust the **CALIB** so that the display indicates the pH of the buffer at the solution temperature. See **Table 1**.
4. Remove the electrode from the buffer solution and rinse by stirring moderately in distilled water. Shake off excess drops of water.
5. Place electrode in the sample to a depth of about 3 cm and stir moderately. Set the function control to ON and allow the reading to stabilize. Record the steady pH reading.

two-buffer standardization
(where maximum precision is required)

1. Select two buffers to bracket the expected pH of the sample, with one buffer having a pH of 7.
2. Place the electrode in the pH 7 buffer to a depth of about 3 cm and stir moderately. Set the **temperature indicator control** to the temperature of the buffer. Set the **function control** to **ON** and allow the reading to stabilize. Turn **CALIB** until the display indicates the pH of the buffer at the solution temperature. See **table 1**.
3. Remove electrode from the first buffer and rinse by stirring moderately in distilled water. Shake off excess drops of water.
4. Place the electrode in the second buffer to a depth of about 3 cm and stir moderately. Set the **function control** to **ON** and adjust the **slope control** until the pH at the solution temperature is displayed. See **Table 1**.
5. Remove the electrode and rinse by stirring moderately in distilled water. Shake off excess drops of water.
6. Place the electrode in the sample to a depth of about 3 cm and stir moderately. Set the **function control** to **ON** and allow the reading to stabilize. Record the steady pH reading.

TABLE 1

TEMP (°C)	pH 7.00 Buffer	pH 4.01 Buffer	pH 10.01 Buffer
5	7.08	4.00	10.25
10	7.06	4.00	10.18
15	7.03	4.00	10.12
20	7.01	4.00	10.06
25	7.00	4.01	10.01
30	6.98	4.02	9.97
35	6.98	4.02	9.93
40	6.97	4.03	9.89
50	6.97	4.06	9.83
60	6.98	4.09	--

battery replacement

To replace the batteries, remove the panel on the back of the meter. Be sure to observe the polarity marking when inserting new batteries.

recorder output

The red and black binding posts at the side of the meter provide an output for strip chart recording of absolute mV independent of function mode. For recorders with input impedance of 100 Kilohms or greater, the output is fixed to about 100 mV/pH. pH 14.00 output is 1.40 V. Lower impedance recorders may be used but full-scale output is reduced.

1. Connect the lead from the high (input side of the recorder) to the red binding post and the lead from the low (ground) side to the black binding post.
2. Proceed according to directions in the strip chart recorder instruction manual.

repair and service

ORION warranty covers failures due to manufacturer's workmanship or material defect from the date of purchase by the user. User should return the warranty card to ORION and retain proof of purchase. Warranty is void if product has been abused, misused, or repairs attempted by unauthorized persons.

Warranties herein are for products sold/installed for use only in the United States and Canada. For ORION products purchased for use in all other countries consult local in-country, authorized ORION sales agent/distributor for product warranty information.

A Return Authorization Number must be obtained from ORION Laboratory Products Customer Service before returning any product for in-warranty repair, replacement or credit.

"No Lemon" Instrument Warranty

The instrument is covered by the ORION "No Lemon" warranty. If the instrument fails within twelve months from date of purchase for any reason other than abuse, the purchaser may elect to have it repaired or replaced at no charge. This warranty covers the original or replacement/repaired meter from date of original meter purchase; the warranty is not extended beyond the buyer's original warranty date.

accessories

815600	Ross™ epoxy body, bulb guard combination pH electrode
9104BN	Laboratory grade combination pH electrode (BNC connector)
910600	GX-series epoxy body, gel-filled combination electrode (BNC connector)
912600	GX-series epoxy body, gel-filled flask combination electrode (BNC connector)
913600	GX-series epoxy body, gel-filled flat surface combination pH electrode (BNC connector)
915600	RX-series refillable, epoxy body combination pH electrode (BNC connector)
9162BN	Combination pH electrode with rugged bulb (BNC connector)
9163BN	Combination pH electrode with needle shape (BNC connector)
910004	pH 4 buffer packets, box of 25 packets, each packet making 200 ml of buffer
910007	pH 7 buffer packets, box of 25 packets, each packet making 200 ml of buffer
910009	pH 9 buffer packets, box of 25 packets, each packet making 200 ml of buffer
910104	pH 4.01 buffer, 475 ml bottle
910107	pH 7.00 buffer, 475 ml bottle
910110	pH 10.01 buffer, 475 ml bottle
970899	Dissolved oxygen electrode
910002	Electrode holder
020030	Shorting plug
020120	110V AC line adapter
020121	220V AC line adapter

specifications

package contents	model 211 digital pH meter, with model 910600 gel-filled unbreakable combination pH electrode, support rod, electrode holder, bottles for pH 7 buffer and distilled water, one packet pH 7 buffer powder, AC adapter, six 1.5 V batteries, and carrying case
range	0 to 14 pH
resolution	± .01 pH
temperature compensation	manual (0 to 100°C)
isopotential point	pH 7 (fixed)
power requirement	six 1.5 V batteries; **battery life:** 3000 ten second intermittent measurements when line adapter is not used. **line adapter:** 110 or 220 V ± 20%, 50/60 Hz
dimensions	14 cm high x 9 cm wide x 4.5 cm deep
weight	0.4 kg

specifications subject to change without notice

notice of compliance

The Model 211 may generate radio frequency energy and if not installed and used properly, that is, in strict accordance with the manufacturer's instructions, may cause interference to radio and television reception. It has been type tested and found to comply with the limits for a Class B computing device in accordance with specifications in Subpart J of Part 15 of FCC Rules, which are designed to provide reasonable protection against such interference in a residential installation. However, there is no guarantee that interference will not occur in a particular installation. If the Model 211 does cause interference to radio or television reception, which can be determined by turning the unit off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- reorient the receiving antenna
- relocate the Model 211 with respect to the receiver
- move the Model 211 away from the receiver
- plug the Model 211 into a different outlet so that the meter and receiver are on different branch circuits

If necessary, the user should consult the dealer or an experienced radio/television technician for additional suggestions. The user may find the following booklet prepared by the Federal Communications Commission helpful:

"How to Identify and Resolve Radio-TV Interference Problems"

This booklet is available from the U.S. Government Printing Office, Washington, DC 20402, Stock No. 004-000-00345-4.

Appendix F
FIELD MEASUREMENT OF SPECIFIC CONDUCTANCE AND TEMPERATURE

FIELD MEASUREMENT OF SPECIFIC CONDUCTANCE AND TEMPERATURE

Method: Specific Conductance, umhos @ 25°C

Reference: EPA 1979, Page 120.1, Standard Methods, 15th edition, pp 70-73

Detection Limit: 1 umho/cm @ 25°C

Optimum Range: 0.1 - 100,000 umhos/cm

Sample Handling: Determine on-site or within 24 hours

Reagents and Apparatus:

1. Conductivity meter (YSI) and electrodes.
2. Deionized water in squirt bottle.
3. Standard potassium chloride solution, 0.0100 N.

Procedure:

YSI Conductivity Meter

1. With mode switch at off position, check meter zero. If not zeroed, use meter screw and adjust to zero.
2. Plug probe into jack on side of meter.
3. Turn mode switch to red line, and turn red line knob until needle aligns with red line on dial. Change batteries if cannot be aligned.
4. Totally immerse probe in sample. Do not allow the probe to touch the sample container.
5. Turn mode switch to appropriate conductivity scale, X100, X10, or X1. Use a scale that will give a mid-range output on the meter.
6. Wait for needle to stabilize (about 15 sec.) and record conductivity multiplying by scale setting.
7. While gently agitating the probe, take sample temperature (°C) and record.
8. Rinse probe with deionized water.
9. Record specific conductivity (1st column) and temperature on F.O.S. sheet.

Notes:

1. Calculate conductivity using following formula:

$$G_{25} = \frac{G_T}{[1 + 0.02\ (T-25)]}$$

G_{25} = Conductivity at 25°C, umhos/cm

T = Temperature of sample, °C

G_T = Conductivity of sample at temperature T, umhos/cm

2. Report results for the standard solution with each data set.

3. Record on field sheet which meter and probe were used. Meter should be wiped clean as necessary.

4. After returning to lab, compare results with previous data. Report problems to lab personnel.

Reagent Preparation:

1. Stock Potassium Chloride Solution, 1.00 N: Dissolve 74.555 g. K Cl in Milli-Q water and dilute to 1,000 ml. in a volumetric flask.

2. Standard Potassium Chloride Solution, 0.0100N: Dilute to 10.0 mls. of stock solution to 1,000 mls. with Milli-Q water using a volumetric pipet and flask.

ATTACHMENT 1

OPERATING INSTRUCTION
YSI MODEL 33
CONDUCTIVITY METER

TABLE OF CONTENTS

GENERAL DESCRIPTION

The YSI Model 33 and 33M S-C-T Meters are portable, battery powered, transistorized instruments designed to accurately measure salinity, conductivity and temperature. They use a probe consisting of a rugged, plastic conductivity cell and a precision YSI thermistor temperature sensor combined in a single unit.

Conductivity with the Model 33 is expressed as micromhos/centimeter (μmhos/cm); with the 33M, it's millisiemens/meter (mS/m). These are measurements of the electrical conductance the sample would show if measured between opposite faces of a 1cm cube. (Conversion information: 1 μmho/cm = 0.1 mS/m.) Salinity is the number of grams of salt/kilogram of sample (‰ = parts per thousand). This measurement assumes the sample contains a "standard" sea water salt mixture. The sample temperature is measured in degrees Celsius.

Salinity measurements are manually temperature compensated by direct dial. Conductivity measurements are not temperature compensated, however, a temperature function is provided on the instrument to aid with calculation of corrections. Also, when just temperature and conductivity are known it is possible to calculate salinity and when only temperature and salinity are known it is possible to calculate conductivity.

SPECIFICATIONS

Model 33 Conductivity

Ranges	0-500, 0-5,000, 0-50,000 μmhos/cm with YSI 3300 Series Probes (Note: The "μmho" designations on the meter are a shorthand form for "μmho/cm".)
Accuracy	±2.5% max. error at 500, 5,000 and 50,000 plus probe ±3.0% max. error at 250, 2,500 and 25,000 plus probe See Error Section

Readability	2.5 μmhos/cm on 500 μmho/cm range 25 μmhos/cm on 5,000 μmho/cm range 250 μmhos/cm on 50,000 μmho/cm range
Temperature Compensation	None

Model 33M Conductivity

Ranges:	0-50, 0-500, 0-5,000 mS/m with YSI 3300 Series Probes
Accuracy:	±2.5% max. error at 50, 500 and 5,000 plus probe ±3.0% max. error at 25, 250 and 2,500 plus probe See Error Section
Readability	0.25 mS/m on 50 mS/m range 2.5 mS/m on 500 mS/m range 25.0 mS/m on 5,000 mS/m range
Temperature Compensation	None

Salinity

Range:	0-40 ‰ in temperature range of -2 to +45°C
Accuracy	Above 4°C: ±0.9 ‰ at 40 ‰ and ±0.7 ‰ at 20 ‰ plus conductivity probe Below 4°C: ±1.1 ‰ at 40 ‰ and ±0.9 ‰ at 20 ‰ plus conductivity probe See Error Section
Readability	0.2 ‰ on 0-40 ‰ range
Temperature Compensation	Manual by direct dial from -2 to +45°C

Temperature	
Range	-2 to +50°C
Accuracy	±0.1°C at -2°C, ±0.6°C at 45°C plus probe See Error Section
Readability	±0.15°C at -2°C to ±0.37°C at 45°C
Power Supply	Two D-size alkaline batteries. Eveready E95 or equivalent provide approximately 200 hrs. of operation
Probe	YSI 3300 Series Conductivity/Temperature Probe Nominal Probe Constant K = 5/cm
Accuracy	±2% of reading for conductivity and salinity Error of ±0.1°C at 0°C and ±0.3°C at 40°C
Instrument	
Ambient Range	Satisfactory operation -5 to +45°C. A maximum error of ±0.1% of the reading per °C change in instrument temperature can occur. This error is negligible if the instrument is readjusted to redline for each reading

OPERATION PROCEDURE

1. **Setup**
   (a) Adjust meter zero (if necessary) by turning the bakelite screw on the meter face so that the meter needle coincides with the zero on the conductivity scale
   (b) Calibrate the meter by turning the MODE control to REDLINE and adjusting the REDLINE control so the meter needle lines up with the redline on the meter face. If this cannot be accomplished, replace the batteries
   (c) Plug the probe into the probe jack on the side of the instrument
   (d) Put the probe in the solution to be measured. (See Probe Use.)
2. **Temperature**
   Set the MODE control to TEMPERATURE. Read the temperature on the bottom scale of the meter in degrees Celsius. Allow time for the probe temperature to come to equilibrium with that of the water before reading.
3. **Salinity**
   (a) Transfer the temperature reading from Step 2 to the °C scale on the instrument.
   (b) Switch the MODE control to the SALINITY position and read salinity on the red 0-40 ‰ meter range.
   (c) Depress the CELL TEST button. The meter reading should fall less than 2%; if greater, the probe is fouled and the measurement is in error. Clean the probe and re-measure.
4. **Conductivity on Model 33 (Model 33M data are in parentheses.)**
   (a) Switch the MODE control to the X100 scale. If the reading is below 50 on the 0-500 range (5.0 on the 0-50 range) switch to the X10 scale. If the reading is still below 50 (5.0), switch to the X1 scale. Read the meter scale and multiply the reading appropriately. The answer is expressed in μmhos/cm (mS/m). Measurements are not temperature compensated.

Example:	Meter Reading	247 (24.7)
	Scale	X10
	Answer	2470 μmhos/cm (24.7 0 mS/m)

(b) When measuring on the X100 and X10 scales, depress the CELL TEST button. The meter reading should fall less than 2%, if greater, the probe is fouled and the measurement is in error. Clean the probe and re-measure.

NOTE: The CELL TEST does not function on the X1 scale

5. **Error**

The maximum error in a reading can be calculated by using the graphs in the following sections.

(1) Temperature

The temperature scale is designed to give the minimum salinity error when the temperature readings are used to compensate salinity measurements.

Figure 1 shows total error for probe and instrument versus °C meter reading.

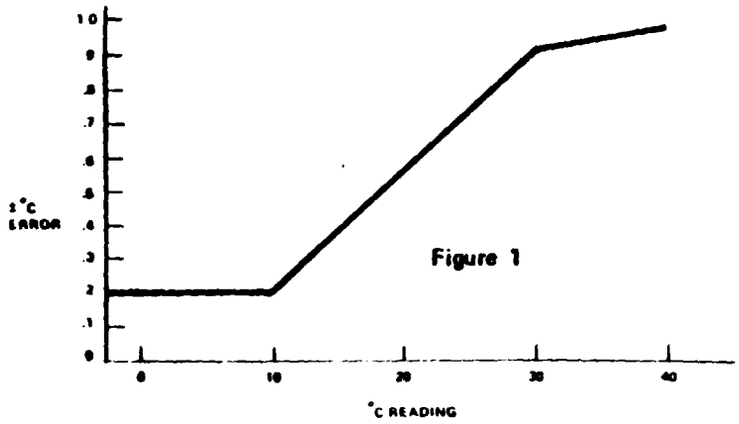


Figure 1

Example: Meter Reading 15°C

Total Error 0.4°C

Accuracy 15°C ± 0.4°C for probe and instrument combined

(2) Conductivity on Model 33 (Model 33M data are in parentheses)

Figure 2 shows the worst-case conductivity error as a function of the conductivity reading for the probe and instrument combined.

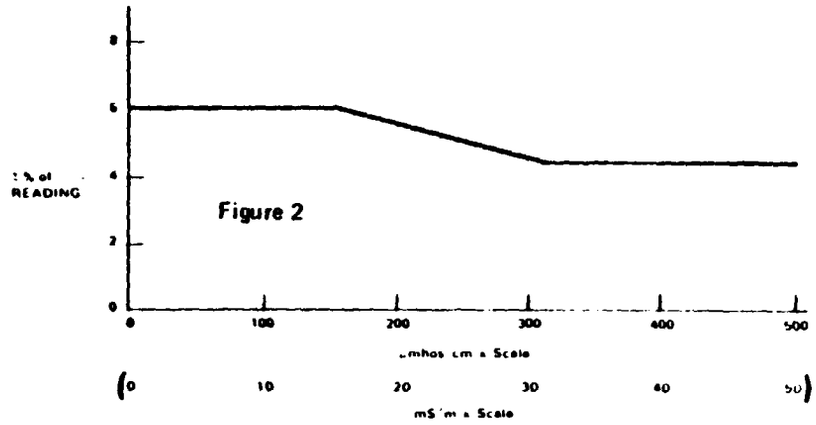


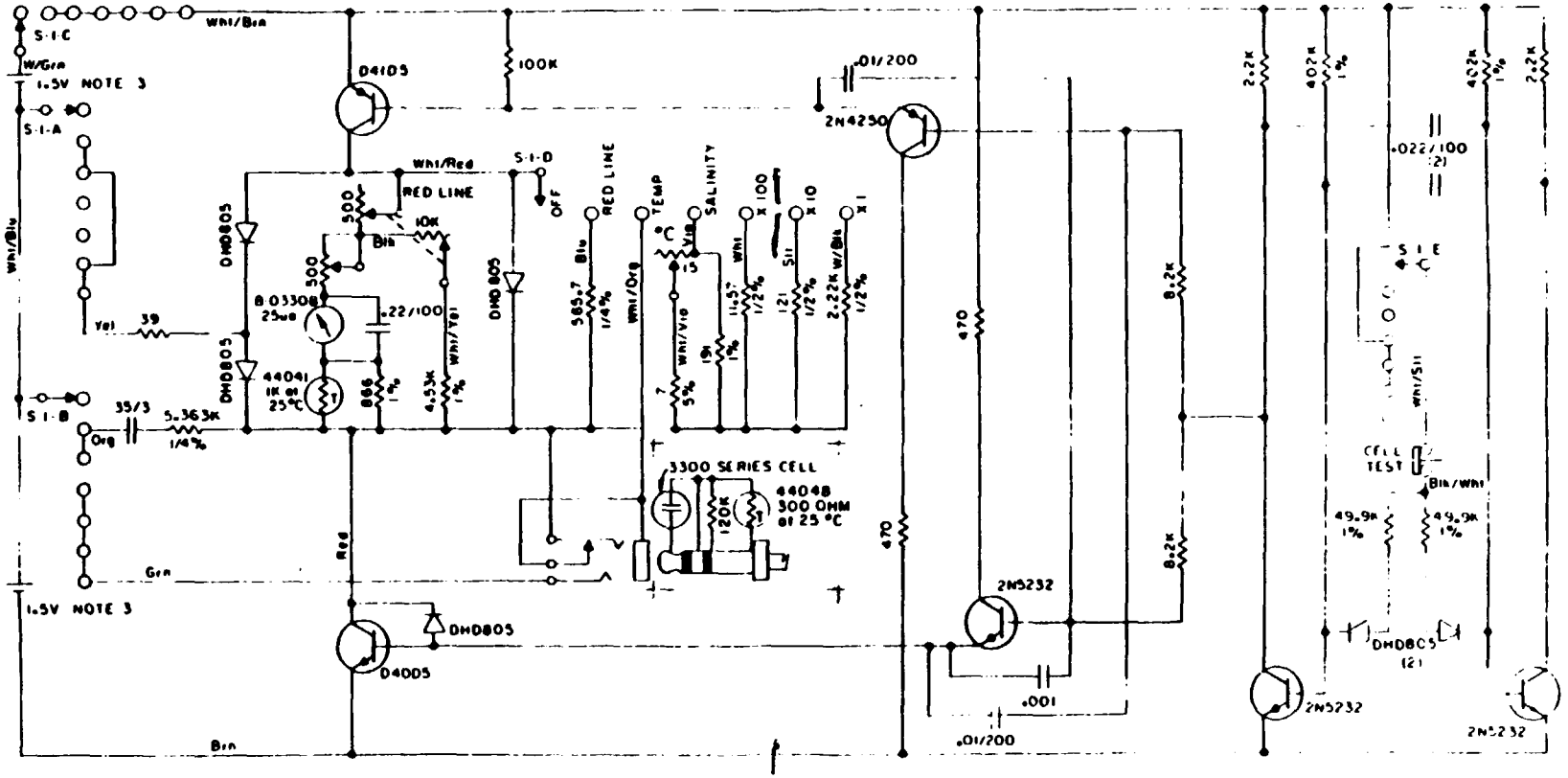
Figure 2

Example: Meter Reading 360 µmhos/cm (36 mS/m)

Scale X10

% Reading Error ± 4.5%

Accuracy 3600 ± 162 µmhos/cm (360 ± 16.2 mS/m) for probe and instrument



NOTE

1. Resistance values in ohms, K=1,000 resistors are ½W, 10% unless otherwise specified
2. The values shown on the schematic may differ from those in the instrument. If so, either value can be used for replacement purposes
3. Battery is "D" size, alkaline only, Eveready E 95 or equal

YSI MODEL 33 AND 33M B-03321-E

CIRCUIT DESCRIPTION, MAINTENANCE AND CALIBRATION

1. Description

The circuit is composed of two parts, a multivibrator and switching transistors. The multivibrator produces a square waveform voltage. The square wave is applied to two switching transistors. They alternately apply two batteries of opposite polarity to the probe thus providing AC power which minimizes polarization effects. The meter is in series with one battery and measures the current from it. The current from the battery is proportional to the conductance of the cell. Salinity is measured in a special range conductivity circuit which includes a user-adjusted temperature compensator. In the temperature, redline and X1 positions the multivibrator operates at 100 Hz. In the salinity, X100 and X10 positions the multivibrator operates at 600 Hz and in these ranges pushing the CELL TEST button drops the frequency to 100 Hz allowing the operator to judge the degree of probe polarization.

2. Maintenance

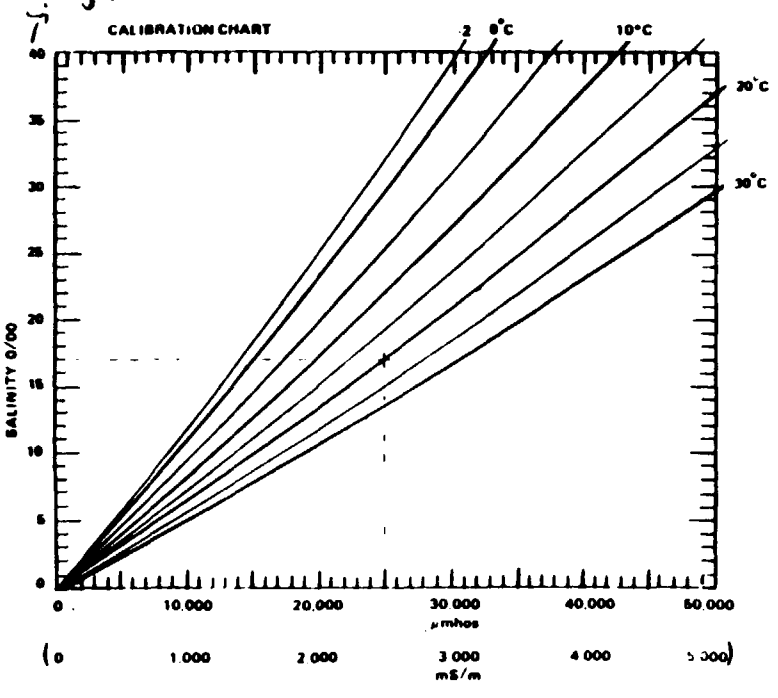
The only maintenance required is battery replacement. Two "D" size alkaline flashlight cells, such as Eveready E95 or equivalent, will provide 200 hrs. of operation. Accuracy will not be maintained if zinc-carbon "D" cells are used. Battery replacement is indicated when the redline adjustment cannot be accomplished.

Replace batteries every six months to reduce the danger of corrosion due to leaky batteries. To replace batteries, remove the six screws from the rear plate. The battery holders are color coded. The Positive (+ button) end must go on red.

3. Calibration of Model 33 (Model 33M data are in parentheses.)

It is possible for the temperature knob to become loose or slip from its normal position. In an emergency the dial can be re positioned. It must be emphasized that this is an emergency procedure only, and that the instrument should be returned to the factory for proper recalibration at the earliest opportunity.

(a) Read the temperature and conductivity of the solution. Determine the salinity of the solution by running a line vertically on the graph from this conductance value until it intersects the appropriate °C line (interpolate as required for temperature between the given °C lines). From this intersection extend a line horizontally to the edge of the graph. This determines the salinity for this sample.
Example: 25,000 μmhos/cm and 20°C gives a salinity of 17.
(Example: 2.500 mS/m and 20°C gives a salinity of 17.)



(b) Remove the °C knob, switch to SALINITY, and turn the control shaft until the meter needle indicates the salinity value determined in Step (a). In the example given, the value is 17.

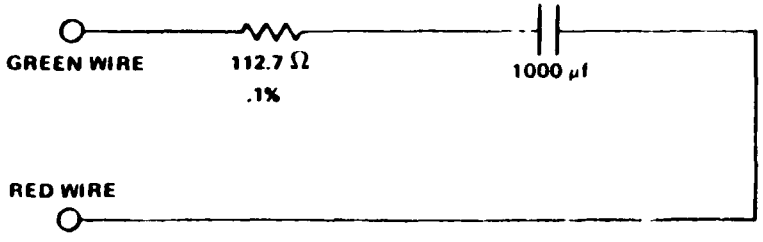
(c) Switch to TEMPERATURE. (Note: This temperature reading must be the same as Step (a), if not, begin again at Step (a).) Place the knob on the control shaft (without turning the control shaft) with the knob pointer at the same temperature as the meter reading and tighten both set screws securely.

At earliest opportunity recalibrate using the following procedure or return the instrument to factory for service.

(a) Set the instrument for a salinity measurement as normal.

(b) Substitute a 1000 μf capacitor and 112.7 ohm 0.1% tolerance resistor for the probe.

Connect the resistor and capacitor between the green wire and red wire on the jack connections inside the instrument.



(c) Turn the temperature dial until the meter reads redline

Now install the temperature knob with the arrow at 25°C This is a temporary calibration only Return the instrument to the factory for proper recalibration

PROBE

1. Description of YSI 3300 Series Conductivity/Temperature Probe

The YSI 3300 Series Conductivity Probes are designed for field use, embodying construction and design for rugged, accurate service

Each probe features a built-in cell constant of 5 0 (500 0/M) ±2%, a precision YSI thermistor temperature sensor of ±0 1°C accuracy at 0°C and ±0 3°C at 40°C and a low capacitance cable assembly terminating in a three therminal 0 25" dia phone type connector.

The 3310 has a 10 ft cable and the 3311 is a 50 ft version Other lengths are available on special order

The probe has a rigid P V C body, platinized pure nickel electrodes, and a durable cable, providing resistance to a wide range of water-borne substances

2. Maintenance

(a) Cleaning

When the cell test indicates low readings the probable cause is dirty electrodes Hard water deposits, oils and organic matter are the most likely contaminants

For convenient normal cleaning soak the electrodes for 5 minutes with a locally available bathroom tile cleaning preparation such as Dow Chemical "Bathroom Cleaner", Horizon Industries "Rally Tile, Porcelain, and Chrome Cleaner", Johnson Wax "Envy Instant Cleaner" or Lysol Brand "Basin, Tub Tile Cleaner"

For stronger cleaning a 5 minute soak in a solution made of 10 parts distilled water, 10 parts isopropyl alcohol and 1 part HCl can be used.

Always rinse the probe after cleaning and before storage

CAUTION: Do not touch the electrodes inside the probe Platinum black is soft and can be scraped off

If cleaning does not restore the probe performance, re platinizing is required.

(b) Re Platinizing

Equipment Required —

(1) YSI #3140 Platinizing Solution, 2 fl oz (3% platinum chloride dissolved in 0 025% lead acetate solution)
(2) YSI Model 33 or 33M S-C-T Meter
(3) 50 ml glass breaker or equivalent bottle
(4) Distilled water

Procedure —

(1) Clean the probe as in Section (a) -- either method
(2) Place the cell in the beaker and add sufficient YSI #3140 solution to cover the electrodes Do not cover the top of the probe
(3) Plug the probe into the Model 33 or 33M, switch to the X100 scale to platinize the electrode Move the probe slightly to obtain the highest meter reading and continue platinizing for the approximate time shown below

Meter Reading		Time
µmhos/cm	mS/m	(minutes)
30,000	3.000	5
25.000	2.500	6
20.000	2.000	8
15.000	1.500	11
10.000	1.000	16

(4) After the elapsed time remove the probe and rinse in fresh water.

(5) Return the solution to its container. 2 oz. of solution should be sufficient for 50 treatments.

3. Probe Use

(a) Obstructions near the probe can disturb readings. At least two inches of clearance must be allowed from non-metallic underwater objects. Metallic objects such as piers or weights should be kept at least 6 inches from the probe.

(b) Weights are attached to the cable of the YSI 3310 and 3311 Probes. The YSI 3327 Weights are supplied in pairs with a total weight of 4 ounces per pair. Should it become necessary to add more weight to overcome water currents, we suggest limiting the total weight to two pounds (8 pairs). For weights in excess of two pounds use an independent suspension cable. In either case, weights must be kept at least 6 inches away from the probe.

(c) Gentle agitation by raising and lowering the probe several times during a measurement insures flow of specimen solution through the probe and improves the time response of the temperature sensor.

4. Cell Calibration & Standard Solutions

The YSI #3300 Series Cells are calibrated to absolute accuracy of ±1.5% based on a standard solution. Since the literature on conductivity does not indicate a consistently accepted standardization method, we have chosen the 0.01 demal KCl solution method as determined by Jones and Bradshaw in 1937 as our standard. Recent textbooks, as well as the ASTM standards, concur with this choice.

The solution is prepared by diluting 0.745 grams of pure dry KCl with distilled water until the solution is 1 kilogram. The table below shows the values of conductivity this solution would have if the distilled water were non-conductive. However, since even high purity distilled

water is slightly conductive, the measured conductivity will be higher by an amount equal to the water's conductivity.

Temperature °C	Conductivity μmhos/cm	mS/m
15	1141.5	114.2
16	1167.5	116.8
17	1193.6	119.4
18	1219.9	122.0
19	1246.4	124.6
20	1273.0	127.3
21	1299.7	130.0
22	1326.6	132.7
23	1353.6	135.4
24	1380.8	138.1
25	1408.1	140.8
26	1436.5	143.7
27	1463.2	146.3
28	1490.9	149.1
29	1518.7	151.9
30	1546.7	154.7

The operator may use the standard solution and the table to check accuracy of a cell's constant or to determine an unknown constant. The formula is shown below

$$K = \frac{R(C_1 + C_2)}{10^6} \quad \text{or} \quad \frac{R(S_1 + S_2)}{10^5}$$

where K = Cell constant
R = Measured resistance in Ω
C_1 = Conductivity in μmhos/cm
C_2 = Conductivity in μmhos/cm of the distilled water used to make solution

S_1 = Conductivity in mS/m
S_2 = Conductivity in mS/m of the distilled water used to make the solution.

R, C_1 and C_2, or S_1 and S_2, must either be determinud at the same temperature or corrected to the same temperature to make the equation valid.

Note: For further information on conductivity and the above standard information, refer to ASTM Standards Part 23 — Standard Methods of Test for Electrical Conductivity, or Water and Industrial Waste Water — ASTM Designation D1125-64.

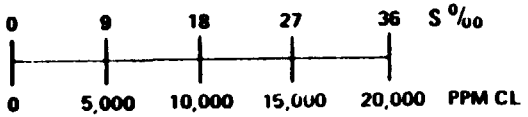
YSI MODEL 33 AND 33M USED WITH YSI 51A, 54 and 57 OXYGEN METERS

If the salinity measurement is to be used for salinity correction on the 51A, the reading should be converted to Chlorosity. The formula is

$$\text{PPM Chlorosity} = \frac{\text{Salinity } ^{o}/_{oo} - 0.03}{1.8} \times 10^{3}$$

For these instruments the 0.03 can be neglected so the equation simplifies to

$$\text{PPM Cl} = \frac{\text{SS } ^{o}/_{oo} \times 10^{3}}{1.8}$$



For salinity correction when using the Model 57 use the salinity reading direct from the Model 33 or 33M. No conversion is necessary.

Model 33 and 33M salinity readings taken in conjunction with Model 54 dissolved oxygen readings can be used to correct the Model 54 for salinity and to make post-measurement salinity corrections to dissolved oxygen data. Correction tables are available from the factory.

WARRANTY

All YSI products carry a one-year unconditional warranty on workmanship and parts, exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge.

If you are experiencing difficulty with any YSI product, it may be returned to an authorized YSI dealer for repair, even if the warranty has expired. If you need factory assistance for any reason, contact

Service Department
Yellow Springs Instrument Co., Inc.
P.O. Box 279
Yellow Springs, Ohio U.S.A.
Phone (513) 767-7241

Appendix G
FIELD MEASUREMENT OF SULFIDE

SULFIDE SPOT TEST

Because of the possible presence of sulfides a spot test for $S^{=}$ will be performed as follows:

> Place a spot of sample on lead acetate test paper previously moistened with acetic acid buffer solution, pH 4 (Std Methods 408B.3e). Darkening of the paper indicates presence of $S^{=}$. The sample will be treated with cadmium carbonate powder. This process will be repeated until the test for sulfide is negative. The sample will then be filtered through a dry glass fiber filter to remove the cadmium sulfide precipitate. The sample will be preserved with saturated NaOH to pH >12.

GLT718/11

Appendix H
FIELD FILTERING OF LIQUID SAMPLES FOR CLP METALS ANALYSIS

FIELD FILTERING OF LIQUID SAMPLES FOR CLP METALS ANALYSIS

Reference: EPA 1979, Metals 5

Sample Handling: Filter as soon as possible after sample collection

Reagents and Apparatus:

1. 10 percent HNO_3 solution in a squirt bottle and in a liter plastic bottle
2. DI water
3. Plastic forceps
4. Filtration apparatus
5. 0.45 um membrane filters (142 mm)
6. Glass fiber prefilters (142 mm)
7. Peristaltic pump

Procedure:

1. Using plastic forceps, place a 0.45 um filter on top of filter apparatus.
2. Place a prefilter on top of membrane filter.
3. Place top onto filter apparatus. Screw wing nut bolts down until even and snug. Finish tightening with plastic wrench.
4. Attach end of PVC hosing from pump to filter apparatus.
5. Run 50-100 mls of HNO_3 through apparatus, rinse with 50-100 mls DI water. Do not collect this filtrate.
6. Place sample bottle under outlet.
7. Turn pump on, run sample through filter, and collect filtered sample from bottom of apparatus.
8. Shut off pump.
9. Rinse twice with DI water, remove filter and dispose, proceed as above for next sample.
10. Run a DI water blank every 10 to 20 samples.

Notes:

1. Samples with high sediment can be filtered through several membranes with increasing pore size and several prefilters. The 0.45 um membrane filter should always be on the grid, and the coarsest filters on the top.

Reagent Preparation:

1. 10 Percent HNO_3 Solution: Add about 900 mls of DI water to a one liter Erlenmeyer flask. Using a graduated cylinder, add 100 mls concentrated HNO_3 to the DI water while stirring.

GLT718/7

Appendix I
INSTRUCTIONS FOR FILLING OUT SAMPLE DOCUMENTATION

INSTRUCTIONS FOR FILLING SAMPLE DOCUMENTATION

All samples collected at Superfund sites for laboratory analysis must follow established documentation protocol. Adherence to this protocol provides a network of valuable information documenting sample identification and tracking as well as chain-of-custody.

GENERAL DOCUMENTATION PROCEDURES

Organization and concentration are the keys to completing the required documents efficiently and without error. Make certain that a suitable work area has been set aside with ample table and floor space available for the processing of forms and the packaging of samples. This is especially important for large projects.

Forms, tags, etc. can be filled out in any order; however, past experience has shown that this paperwork can be completed most efficiently and accurately if the sample identification matrix (Figure 1) is completed before or in conjunction with the completion of the rest of the documentation.

Subsequent sections discuss the proper completion of each document. Use these pages as a reference while following this suggested plan of attack:

1. Make or obtain a list of the samples to be packaged and shipped on the same day and the laboratories to be used.

2. Enter the case number, CRL number, matrix, sample numbers, laboratory, date sampled, and date shipped for each sample on the matrix.

   Note: If portions of a given sample are to be shipped to different laboratories (for organic and inorganic analysis for instance), two entry lines will be required for that sample number to accommodate the chain-of-custody record, airbill, and traffic report numbers corresponding to each portion of the sample.

3. Obtain the QC lot numbers of the prelabeled containers for each sample and enter these on the matrix.

4. Determine the number of shipping containers (coolers) required to accommodate the day's shipment. This is based on the number of samples to be shipped, the number of containers per sample, the number of sample containers that will fit in each cooler, and the number of laboratories to be used.

CH2M HILL
REM/FIT
SAMPLE IDENTIFICATION MATRIX

CASE NUMBER	CRL NUMBER	Matrix	SAMPLE NUMBER	OTR OR HHTR	ITR	CHAIN OF CUSTODY	LAB	DATE SAMPLED	DATE SHIPPED	AIRBILL NUMBER	SAMPLE TAG NUMBERS		QC LOT NUMBERS	

SITE NAME ____ JOB NUMBER ____

NOTE: For purposes of illustration this form has been reproduced at 50% of original size.

FIGURE 1

Note: A group of containers for a single sample should not be split between coolers except when one portion of the sample is to be sent to one laboratory for one type of analysis and the other portion is to be sent to another laboratory for another type of analysis.

5. Complete an airbill for each laboratory address. (Note: Several coolers may be shipped to the same address under one airbill.) Shipment of medium and high concentration samples requires the use of a special airbill, including a shipper's certification for restricted articles (see Figure 12 for example).

6. Enter the airbill numbers on the matrix.

7. Assign a chain-of-custody record to each cooler and determine which sample containers will be shipped in each.

   Note: More than one chain-of-custody record may be needed to accommodate the number of samples to be shipped in one cooler.

8. Assign chain-of-custody numbers to each sample by entering these numbers on the matrix. (Reminder: Portions of samples for organic and inorganic analysis will usually be sent to separate laboratories. Use one line on the matrix for the organics portion information and another line for the inorganics portion information.)

9. If the samples are being shipped under a routine analytical service (RAS), determine the number of organics and/or inorganics traffic reports that will be needed. If the samples are high concentration, determine the number of high hazard traffic reports that will be needed.

10. Assign traffic report numbers to each sample and enter these numbers on the matrix.

11. Assign tag numbers to each sample container for each sample and enter these numbers on the matrix.

12. Complete traffic reports (of SAS packing lists or CRL basic data sheets) based on the information provided on the matrix.

13. Complete sample tags based on the information provided on the matrix and the parameters of analysis. Place tags in groups by sample number.

14. Complete chain-of-custody records based on the information provided on the matrix.

15. Assign two custody seals to each cooler. Enter the serial numbers of the seals in the "REMARKS" section of each chain-of-custody form and temporarily clip seals to the form.

16. Group all the paperwork associated with each cooler in a separate clip.

17. Obtain full signatures of the STL and initials of significant field team members (including yourself) on the sample tags and at the top of the chain-of-custody forms.

18. Prepare to package samples for shipment.

Following are step-by-step instructions for completing each form. The sample identification code to be used is the sample number as described in Appendix A. Other items should be self-evident from the instructions.

SAMPLE IDENTIFICATION MATRIX (FIGURE 1)

1. Indicate site name.

2. Indicate project number.

3. Enter the case number.

4. Enter the CRL number.

5. Specify the sample matrix using the two digit codes listed below followed by the letter (L, M, or H) to indicate low, medium, or high concentrations:

   - o MW--Monitoring Well
   - o LT--Leachate Tank

6. Indicate the sample number.

7. Enter the inorganics traffic report number.

8. Enter the organics traffic report number.

9. Indicate the chain-of-custody report number.

10. Indicate the laboratory to be doing the analysis.

11. Enter the date the sample was taken: month, day, year (no hyphen or slash, e.g., 051284).

12. Enter the shipping date.

13. Enter the airbill number of the shipment.

14. List sample tag numbers corresponding to sample containers shipped under the traffic report number listed in either box 7 or 8.

15. List the QC lot numbers of the containers matching the tag numbers listed in Item 14.

Note: Date recorded on this form must be suitable for computer entry. Each entry must be left justified and must not exceed the number of digits allowed in each section. If portions of samples are to be sent to more than one laboratory for analysis, allow an entire line for each laboratory to accommodate for the additional traffic report, chain-of-custody, and airbill numbers.

SAMPLE TAG (FIGURE 2)

1. Enter the first six digits of the CRL sample identification.

2. Enter the last three digits of the CRL identification code.

3. Enter date of sampling.

4. Enter time of sampling (military time only).

5. Specify "grab" or "composite" sample with an "X."

6. Insert sample identification code.

7. Obtain signature of sample team leader.

8. Indicate presence of preservative with an "X."

9. Specify all parameters for analysis with an "X" for each one.

10a. Indicate traffic report type and serial number (e.g., ITR number: MS 1534).

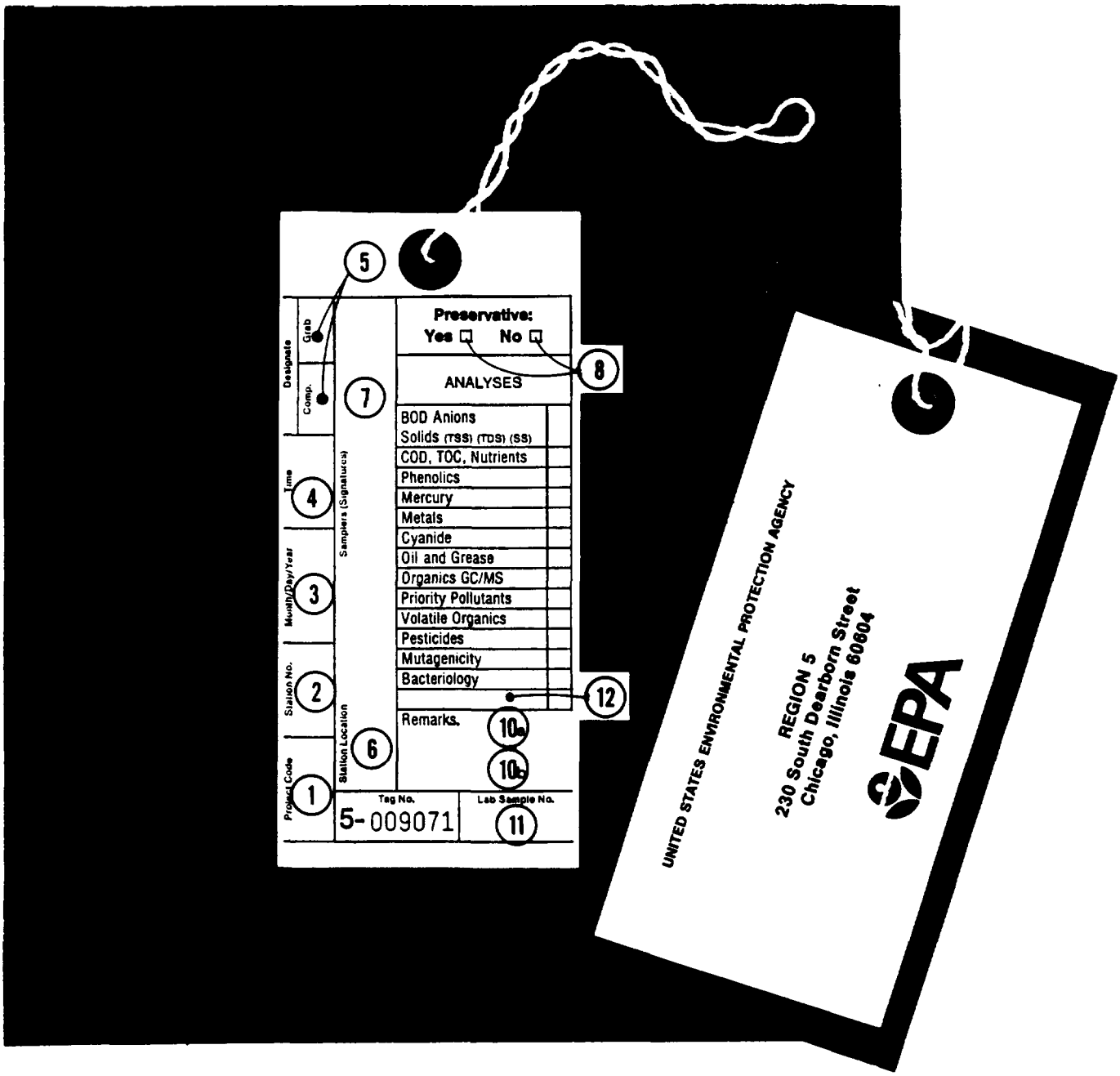
10b. Indicate case number (e.g., CASE No.: 1234).

11. Leave BLANK (for laboratory use only).

12. Enter any desired analyses not listed on menu provided (e.g., PCB's, ammonia, sulfide, etc.) and mark box with an "X."

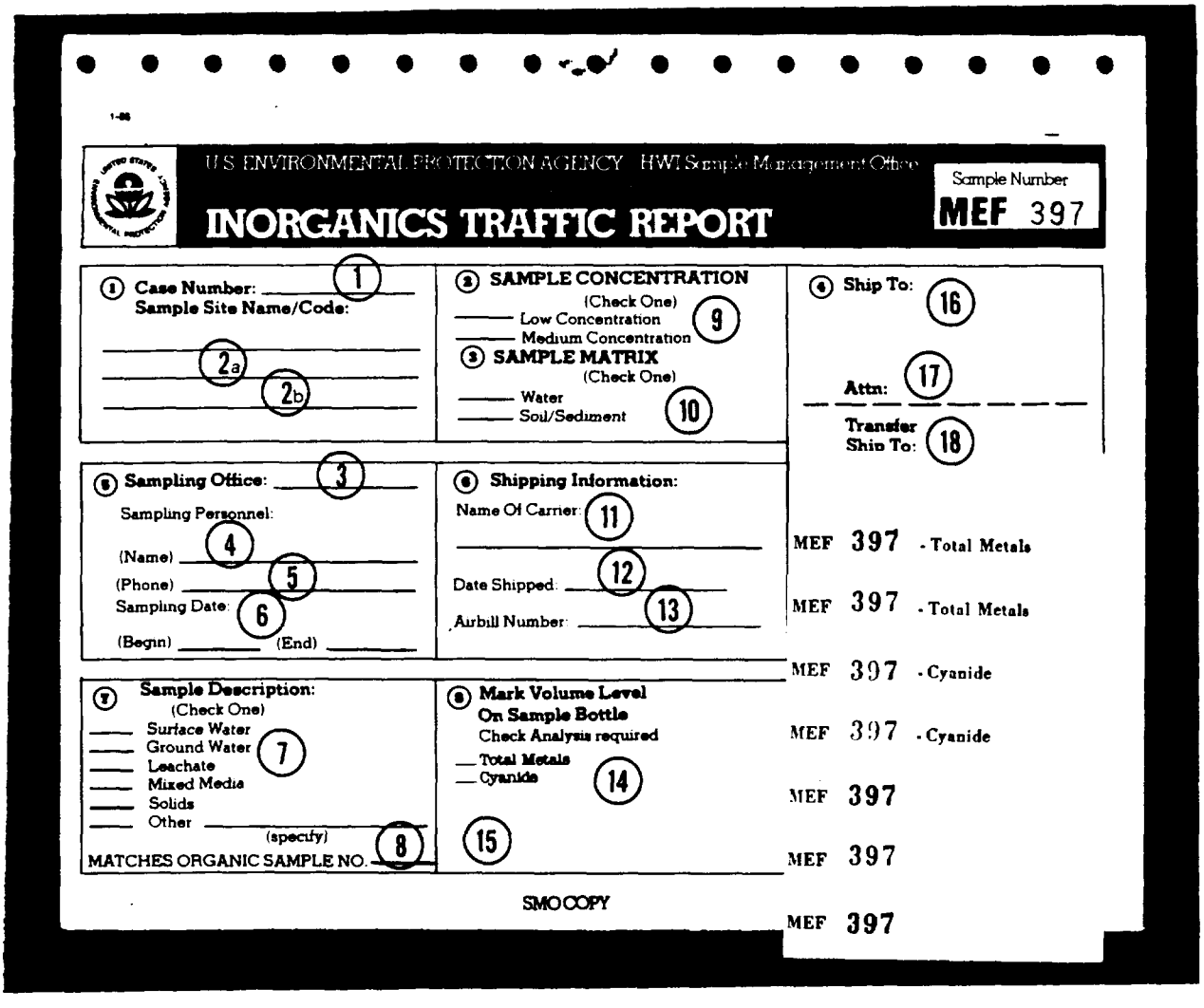
INORGANIC TRAFFIC REPORT (FIGURE 3)

1. Insert assigned laboratory case number.



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 2



U.S. ENVIRONMENTAL PROTECTION AGENCY HWI Sample Management Office

INORGANICS TRAFFIC REPORT

Sample Number MEF 397

(1) Case Number: _____ (1)
Sample Site Name/Code:
_____ (2a)
_____ (2b)
_____

(2) SAMPLE CONCENTRATION
(Check One)
_____ Low Concentration (9)
_____ Medium Concentration
(3) SAMPLE MATRIX
(Check One)
_____ Water
_____ Soil/Sediment (10)

(4) Ship To: (16)

Attn: (17)

Transfer Ship To: (18)

(5) Sampling Office: _____ (3)
Sampling Personnel:
(Name) _____ (4)
(Phone) _____ (5)
Sampling Date: (6)
(Begin) _____ (End) _____

(6) Shipping Information:
Name Of Carrier: (11)
_____
Date Shipped: _____ (12)
Airbill Number: _____ (13)

(7) Sample Description:
(Check One)
_____ Surface Water
_____ Ground Water (7)
_____ Leachate
_____ Mixed Media
_____ Solids
_____ Other _____ (specify)
MATCHES ORGANIC SAMPLE NO. _____ (8)

(8) Mark Volume Level On Sample Bottle
Check Analysis required
__ Total Metals
__ Cyanide (14)

(15)

SMO COPY

MEF 397 - Total Metals
MEF 397 - Total Metals
MEF 397 - Cyanide
MEF 397 - Cyanide
MEF 397
MEF 397
MEF 397

NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 3

2a. Insert CRL sample identification number.

2b. Insert sample number.

3. Insert U.S. EPA region number (e.g., V).

4. Insert sample team leader's name.

5. Insert sample team leader's office telephone number (do not use field office telephone number).

6. Insert date sample was taken.

7. Indicate sample description with an "X."

8. Insert corresponding organic traffic report number for the sample (if any).

9. Specify sample concentration with an "X."

10. Indicate sample matrix with an "X."

11. Insert "Federal Express" (or other approved carrier).

12. Indicate date of shipment.

13. Indicate airbill number corresponding with the sample shipment.

14. Check required analyses: Tasks 1 and 2 (metals) and/or Task 3 (cyanide only, ammonia and sulfide are no longer RAS, although some older traffic reports may still list them).

15. Insert the phrase "QC lot number:" and indicate the quality control lot number(s) of the container(s).

16. Insert laboratory name and address.

17. Indicate name of laboratory contact.

18. Leave BLANK (for laboratory use only).

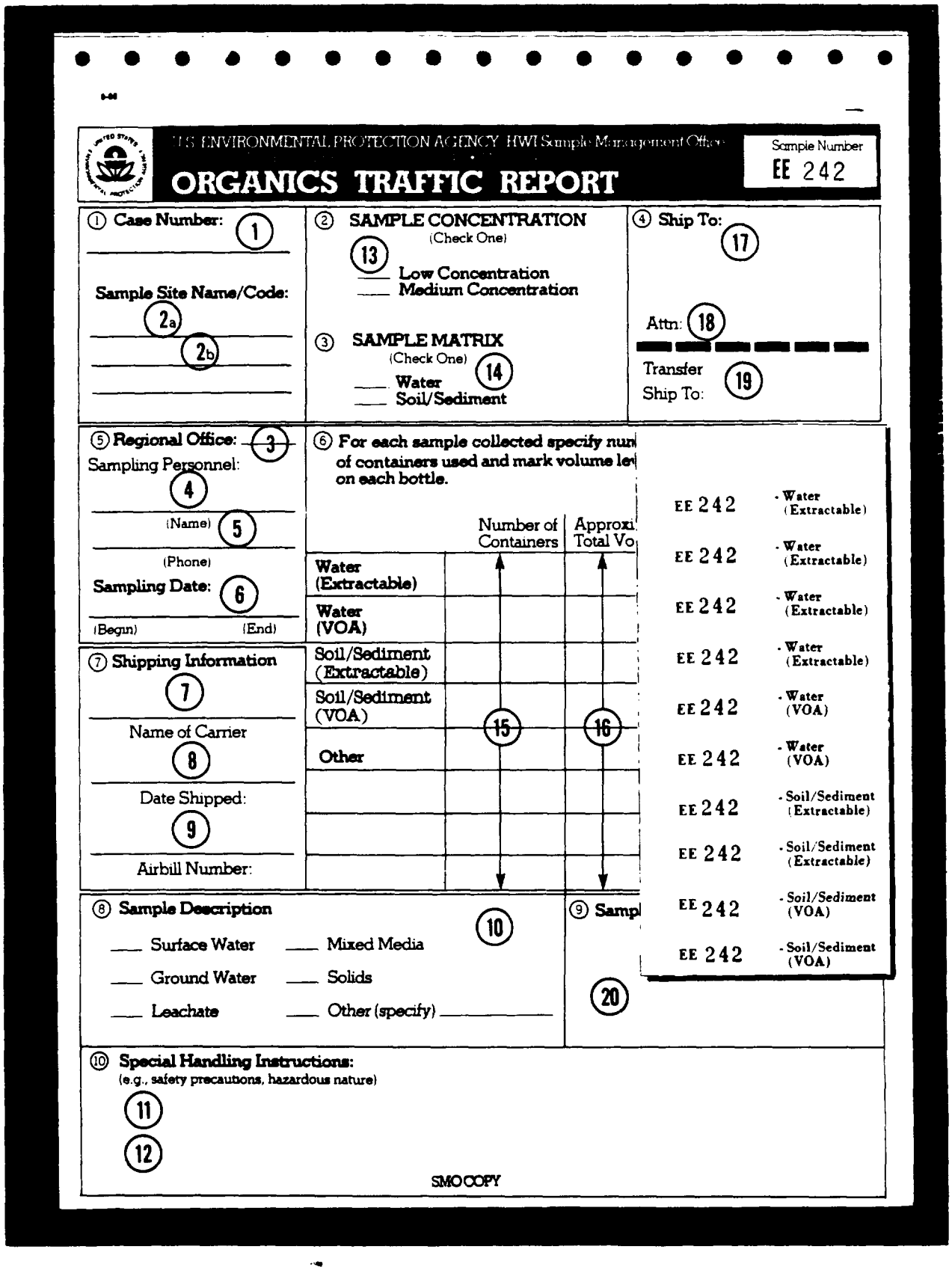
ORGANIC TRAFFIC REPORT (FIGURE 4)

1. Insert assigned laboratory case number.

2a. Insert CRL sample identification number.

2b. Insert sample number.

3. Insert U.S. EPA region number (e.g., V).



U.S. ENVIRONMENTAL PROTECTION AGENCY HWI Sample Management Office

Sample Number EE 242

ORGANICS TRAFFIC REPORT

① **Case Number:** (1)

Sample Site Name/Code:
(2a)
(2b)

② **SAMPLE CONCENTRATION** (13)
(Check One)
___ Low Concentration
___ Medium Concentration

③ **SAMPLE MATRIX** (14)
(Check One)
___ Water
___ Soil/Sediment

④ **Ship To:** (17)

Attn: (18)

Transfer Ship To: (19)

⑤ **Regional Office:** (3)

Sampling Personnel: (4)

(Name) (5)

(Phone)

Sampling Date: (6)

(Begin) (End)

⑥ **For each sample collected specify num of containers used and mark volume le on each bottle.**

	Number of Containers	Approx. Total Vo
Water (Extractable)		
Water (VOA)		
Soil/Sediment (Extractable)		
Soil/Sediment (VOA)	(15)	(16)
Other		

⑦ **Shipping Information** (7)

Name of Carrier (8)

Date Shipped: (9)

Airbill Number:

EE 242	- Water (Extractable)
EE 242	- Water (Extractable)
EE 242	- Water (Extractable)
EE 242	- Water (Extractable)
EE 242	- Water (VOA)
EE 242	- Water (VOA)
EE 242	- Soil/Sediment (Extractable)
EE 242	- Soil/Sediment (Extractable)
EE 242	- Soil/Sediment (VOA)
EE 242	- Soil/Sediment (VOA)

⑧ **Sample Description** (10)

___ Surface Water ___ Mixed Media
___ Ground Water ___ Solids
___ Leachate ___ Other (specify) _____

⑨ **Sampl** (20)

⑩ **Special Handling Instructions:**
(e.g., safety precautions, hazardous nature)
(11)
(12)

SMO COPY

NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 4

4. Insert sample team leader's name.

5. Insert sample team leader's office telephone number (do not use field office telephone number).

6. Insert date sample was taken.

7. Indicate "Federal Express" (or other approved carrier).

8. Indicate date of shipment.

9. Indicate airbill number corresponding to sample shipment.

10. Specify sample description with an "X."

11. Insert the phrase "QC lot number:" and indicate the quality control lot number(s) of the container(s).

12. Insert the phrase "matches IRT number:" and indicate the corresponding inorganics traffic report for the sample (if any).

13. Specify the sample concentration with an "X."

14. Indicate the sample matrix with an "X."

15. Indicate the number of sample containers shipped.

16. Insert an estimated sample volume in appropriate box.

17. Insert laboratory name and address.

18. Indicate name of laboratory contact.

19. Leave BLANK.

HIGH HAZARD TRAFFIC REPORT (FIGURE 5)

1. Insert assigned laboratory case number.

2a. Insert CRL sample identification number.

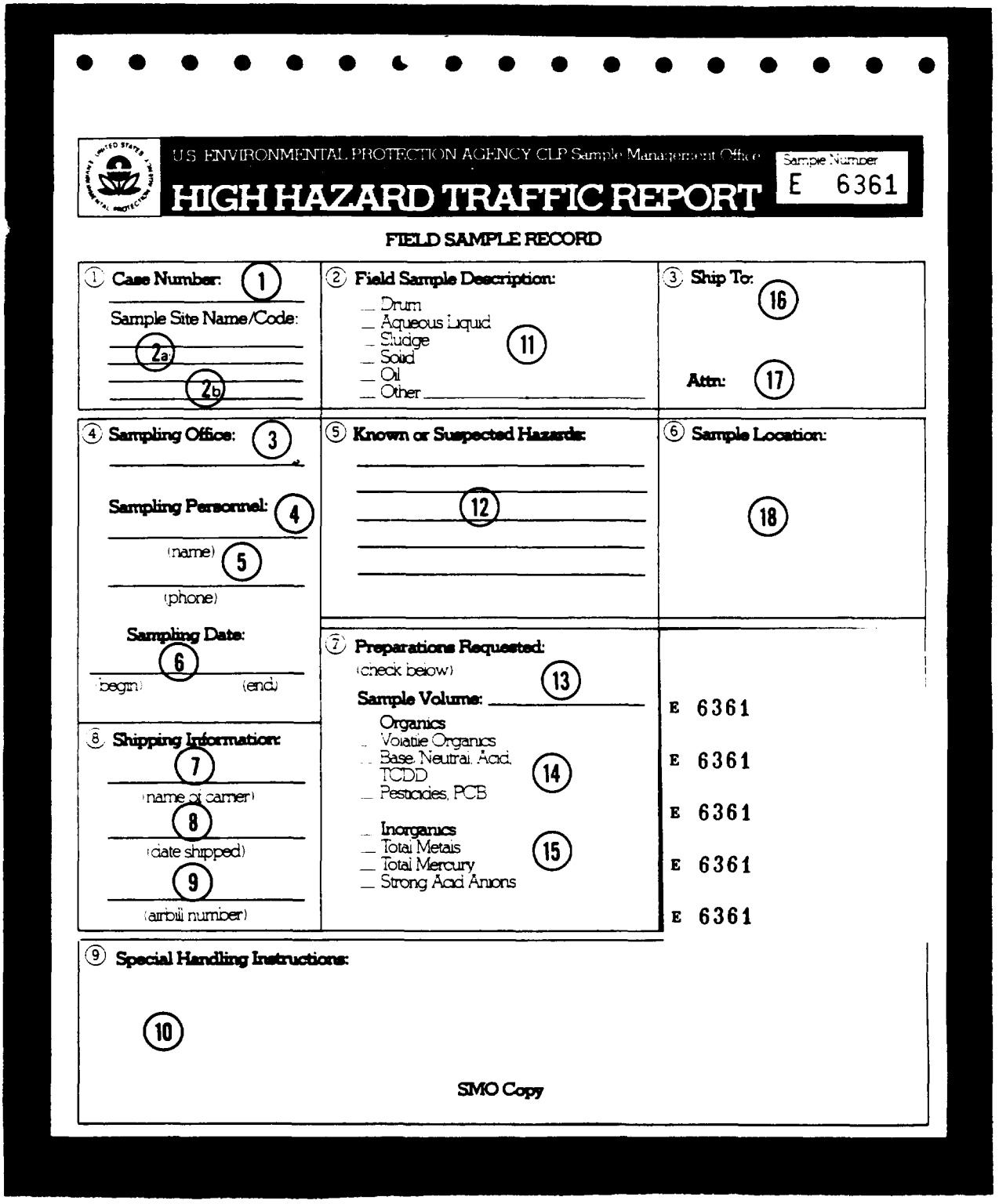
2b. Insert sample number.

3. Insert U.S. EPA region number (e.g., V).

4. Insert sample team leader's name.

5. Insert sample team leader's office telephone number (do not use field office telephone number).

6. Insert date sample was taken.



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 5

7. Indicate "Federal Express" (or other approved carrier).

8. Indicate date of shipment.

9. Indicate airbill number corresponding to sample shipment.

10. Insert the phrase "QC lot number:" and indicate the quality control lot number(s) of the container(s).

11. Indicate sample descriptions with an "X."

12. List known or suspected hazards.

13. Indicate approximate volume of sample.

14. Specify desired organic parameters to be analyzed for.

15. Specify desired inorganic parameters to be analyzed for (strong acid anions include Cl, SO_4, NO_3, F).

16. Insert laboratory name and address.

17. Indicate name of laboratory contact.

18. Leave BLANK (or make reference notes for future use).

SAS PACKING LIST (FIGURE 6)

1. Insert assigned SAS case number.

2. Insert U.S. EPA region number (e.g., V).

3. Insert sample team leader's name.

4. Insert sample team leader's office telephone number (do not use field office telephone number).

5. Insert date sample was taken.

6. Indicate date of shipment.

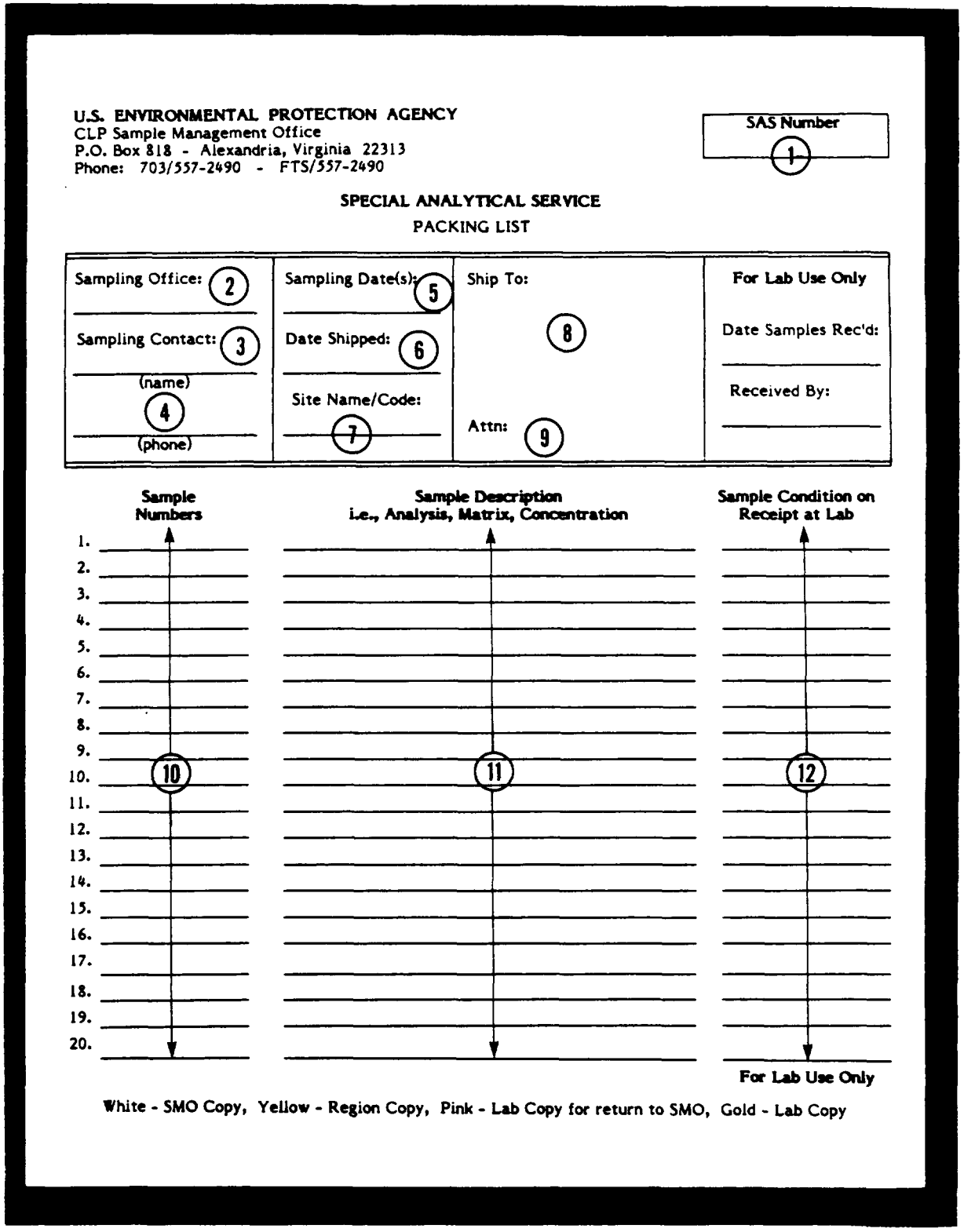
7. Insert site name.

8. Insert laboratory name and address.

9. Indicate name of laboratory contact.

10. List SAS sample numbers, which should include the SAS number.

11. Specify sample matrix, concentration, tag number, and analysis to be performed (e.g., low concentration soil sample for PCB analysis, tag number 5-48246).



U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number (1)

SPECIAL ANALYTICAL SERVICE

PACKING LIST

Sampling Office: (2)	Sampling Date(s): (5)	Ship To: (8)	For Lab Use Only
Sampling Contact: (3)	Date Shipped: (6)		Date Samples Rec'd:
(name) (4) (phone)	Site Name/Code: (7)	Attn: (9)	Received By:

	Sample Numbers (10)	Sample Description i.e., Analysis, Matrix, Concentration (11)	Sample Condition on Receipt at Lab (12)
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			
19.			
20.			
			For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 6

12. Leave BLANK (for laboratory use only).

CHAIN-OF-CUSTODY FORM (FIGURE 7)

1. Enter first six digits of the CRL sample identification code.

2. Enter site code and CH2M HILL project number.

3. Obtain fill signature of sample team leader and signed initials of active team members (including paperwork person).

4. Enter last three digits of the CRL sample identification code.

5. List sampling dates for all samples.

6. List sampling times for all samples.

7. Indicate "grab" or "composite" sample with an "X."

8. List sample numbers.

9. Enter number of containers per sample and container volume (e.g., 2-40 ml).

10. List analyses individually.

11. Construct column heading for traffic report number and list serial numbers for corresponding sample identification codes.

12. Construct column heading for "tag number" and list tag numbers for each sample container.

13. Obtain signature of sample team leader and carry out chain-of-custody procedures.

14. State carrier service and airbill number, lab service, and custody seal numbers.

15. Write in the words "CASE No.:" and enter the case number.

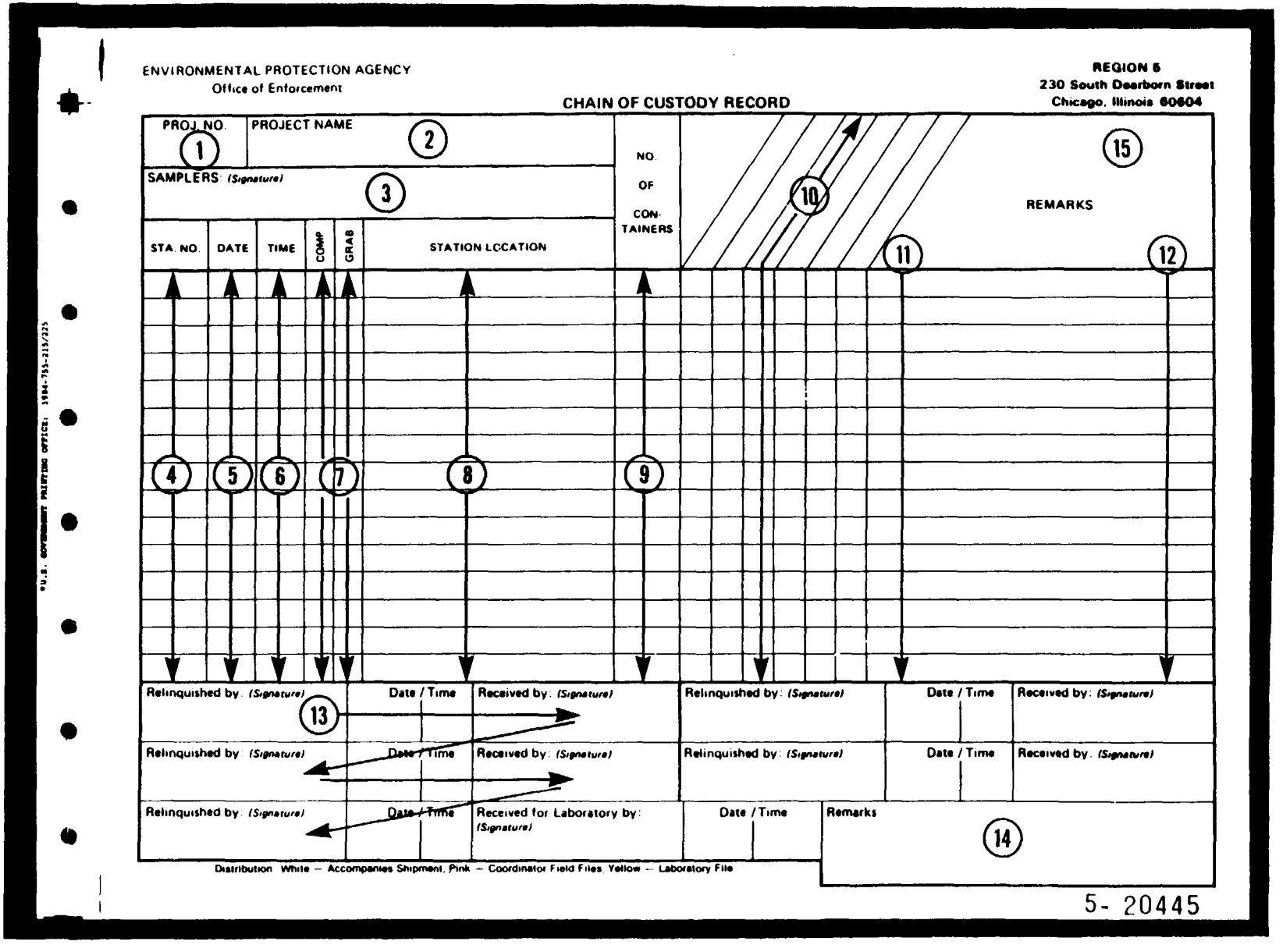
NOTICE OF TRANSMITTAL (FIGURE 8)

1. Enter name of team leader.

2. Enter team leader's firm name.

3. Enter case number.

4. Complete date.



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 7

Figure 8

NOTICE OF TRANSMITTAL

DATE:

TO: CH2M HILL, REM/FIT OFFICE, Region V-X (WI)
310 West Wisconsin Avenue, Suite 700
P.O. Box 2090
Milwaukee, Wisconsin 53201

Attention: Shirley Stringer

FROM: (1) Name / (2) Firm

CH2M HILL PROJECT NO.:

Enclosed are appropriate copies of the sample documentation forms completed under Case #(3) for the (4), 19(4), shipment of (5) Quantity (6) Matrix samples from the (7) site located in (8), (8).

GLT718/9

5. Enter number of samples shipped.

6. Enter matrix of samples.

7. Enter the site name in words.

8. Enter the site location of the site (city, state).

RECEIPT FOR SAMPLES FORM

A completed Receipt for Samples Form will be used whenever splits are provided to other parties. This form must be completed and a copy given to the other party. The original will be retained in the project files. At potential source sites, splits of all samples collected must be offered to an official at the site. If the splits are declined, the Receipt for Samples Form should be so marked.

FIELD LOG BOOK

All information pertinent to a field survey or sampling effort will be recorded in a log book or equivalent standardized form. Each page/form will be consecutively numbered and will be at least 4-1/2 x 7 inches in size. All entries will be made in indelible ink or hard lead pencil and all corrections will consist of line-out deletions that are initialed and dated. As a minimum, entries in a log book will include the following:

- Purpose of sampling.
- Location, description, and log photographs of the sampling point.
- Details of the sampling site (for example, the elevation of the casing, casing diameter and depth, integrity of the casing, etc.).
- Name and address of field contact.
- Documentation of procedures for preparation of reagents or supplied which become an integral part of the sample (e.g., filters and absorbing reagents).
- Identification of sampling crew members.
- Type of sample (for example, groundwater, soil, sludge, or wastewater).
- Suspected waste composition.
- Number and volume of sample taken.

- Sampling methodology, including distinction between grab and composite samples.
- Sample preservation.
- Date and time of collection.
- Collector's sample identification number(s).
- Sample distribution and how transported (for example, name of the laboratory and cartage agent--Federal Express, United Parcel Service).
- References such as maps of the sample site.
- Any field measurements made (for example, pH, specific conductance, temperature, and water depth).
- Signature and date by the personnel responsible for observations.
- Decontamination procedures.

Sampling situations vary widely. No general rules can specify the extent of information that must be entered in a log book or standardized form. However, records will contain sufficient information so that someone can reconstruct the sampling activity without relying on the sample collector's memory. The log book and standardized forms will be kept under strict chain-of-custody.

CORRECTIONS TO DOCUMENTATION

Unless prohibited by weather conditions, all original data recorded on Traffic Report Forms, Sample Identification Tags, Chain-of-Custody Records, and Receipt for Sample Forms will be written with waterproof ink. No accountable serialized documents are to be destroyed or thrown away, even if they are illegible or contain inaccuracies that require a replacement document.

If an error is made on an accountable document assigned to one individual, that individual shall make corrections by making a line through the error and entering the correct information. The erroneous information should not be obliterated. Any subsequent error discovered on an accountable document should be corrected by the person who made the entry. All subsequent corrections must be initialed and dated.

LABORATORY CUSTODY

Laboratory custody will conform to procedures established for the CLP. These procedures include:

- Designation of a sample custodian.
- Correct completion by the custodian of the chain-of-custody record, sample tag, and laboratory request sheet (including documentation of sample condition upon receipt).
- Laboratory sample tracking and documentation procedures.
- Secure sample storage (of the appropriate environment--refrigerated, dry, etc.).
- Proper data logging and documentation procedures including custody of all original laboratory records.

CENTRAL REGIONAL LABORATORY SAMPLE DATA REPORT (FIGURE 9)

The Central Regional Laboratory Sample Data Report form is filled out by the CH2M HILL Sample Documentation Coordinator. A separate report is filled out for each laboratory that receives samples.

1. Enter the case number and/or SAS number.
2. Enter site name.
3. Enter the laboratory name.
4. Enter the date shipped.
5. Enter the Superfund D.U. number.
6. Enter the U.S. EPA RPM.
7. Enter the CERCLIS number.
8. Enter the page numbers.
9. Enter the CRL numbers.
10. Enter the organic or inorganic traffic report number or the SAS packing list number.
11. Check the appropriate box for the analyses to be performed.

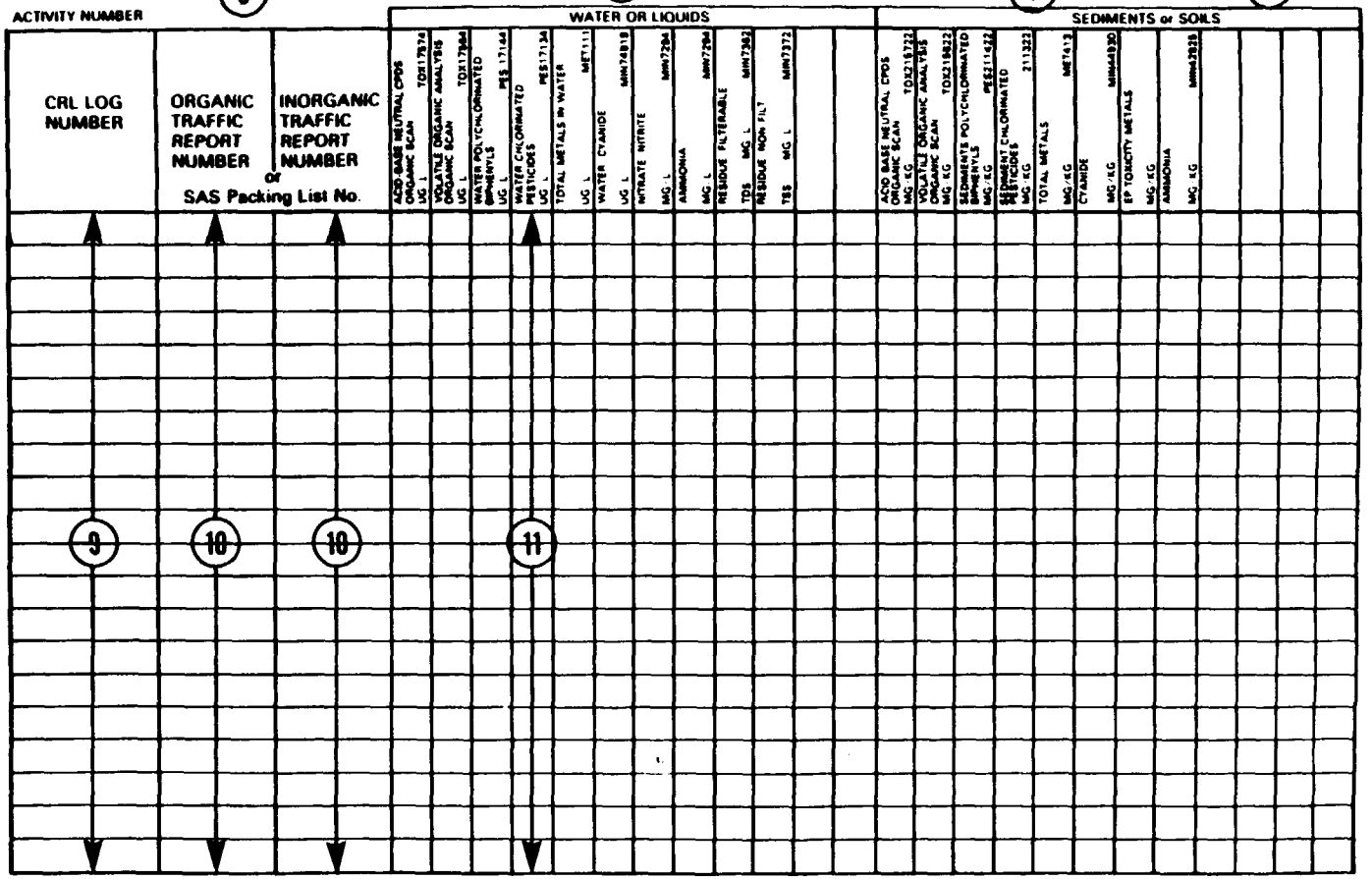
CENTRAL REGIONAL LABORATORY SAMPLE DATA REPORT
ORGANICS/INORGANICS

THIS FORM IS TO BE USED FOR SAMPLES SENT TO CONTRACT ONLY

CASE NUMBER/SAS No. (1) SITE NAME (2) LABORATORY (3) DATE SHIPPED (4)

SUPERFUND DU NUMBER (5) EPA RPM or OSC (S M S)/(CES) (6) CERCLIS NUMBER (7) PAGE (8) OF

ACTIVITY NUMBER



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 9

PACKING AND SHIPPING PROCEDURES

Sample packaging and shipping procedures are based on U.S. EPA Specifications, as well as Department of Transportation (DOT) regulations (40 CFR). The procedures vary according to sample concentration and matrix and are designed to provide optimum protection of samples and the public.

All samples will be shipped within 48 hours of collection or before 50 percent of the holding time has elapsed. Shipping containers must be insulated, durable, and watertight. Bagged samples (to prevent vermiculite contamination of samples, all containers regardless of size/type must be placed inside sealed plastic bag before packing in vermiculite/ zonolite) are to be cushioned within the shipping container with vermiculite packing material (Zonolite). Preformed poly-foam cooler liners are available for shipment of low-concentration samples only.

Following shipment, airbill numbers must be called in to the SMO and to the sample documentation coordinator.

Step-by-step packing instructions are provided below.

LOW-CONCENTRATION SAMPLES

1. Prepare cooler(s)d for shipment.

    - o Tape drain(s) shut.
    - o Affix "This Side Up" labels on all four sides and "Fragile" labels on at least two sides of each cooler.
    - o Place mailing label with laboratory address on top of cooler(s).
    - o Fill bottom of cooler(s) with about 3 inches of vermiculite or use preformed poly-foam liner (low concentration only).
    - o Place appropriate traffic reports, SAS packing lists, or Regional field sheets and chain-of-custody records with corresponding custody seals on top of each cooler.

2. Arrange decontaminated sample containers in groups by sample number.

3. Mark volume levels on bottles with a grease pencil.

4. Secure appropriate sample tags around caps/lids of containers with string or wire.

5. Secure container caps/lids with strapping tape.

6. Arrange containers in front of assigned coolers.

7. Affix appropriate adhesive labels from assigned traffic report to each container. Protect with clear label protection tape.

8. Seal each container within a separate plastic bag.

9. Arrange containers in coolers so that they do not touch.

10. If ice is required to preserve the samples, cubes should be repackaged in double zip-loc bags, and placed on and around the containers (especially on VOA vials).

11. Fill remaining spaces with vermiculite (or place polyfoam liner cover on top of samples).

12. Sign chain-of-custody form (or obtain signature) and indicate the time and date it was relinquished to Federal Express, Purolator, Emery, or other carrier as appropriate.

13. Separate copies of forms. Seal proper copies within a large zip-loc bag and tape to inside lid of cooler. Distribute remaining copies as indicated in the following sections.

14. Close lid and latch.

15. Carefully peel custody seals from backings and place intact over lid openings (right front and left back). Cover seals with clear protection tape.

16. Tape cooler shut on both ends, making several complete revolutions with strapping tape (do not cover custody seals). See Figure 10 for an illustration of a cooler ready for shipment.

17. Relinquish to Federal Express. Place airbill receipt inside the mailing envelope and send to the sample documentation coordinator, along with the other documentation.

18. Telephone the Sample Management Office in Alexandria, Virginia. (Note: This step should be omitted for samples sent to the CRL or outside laboratories).

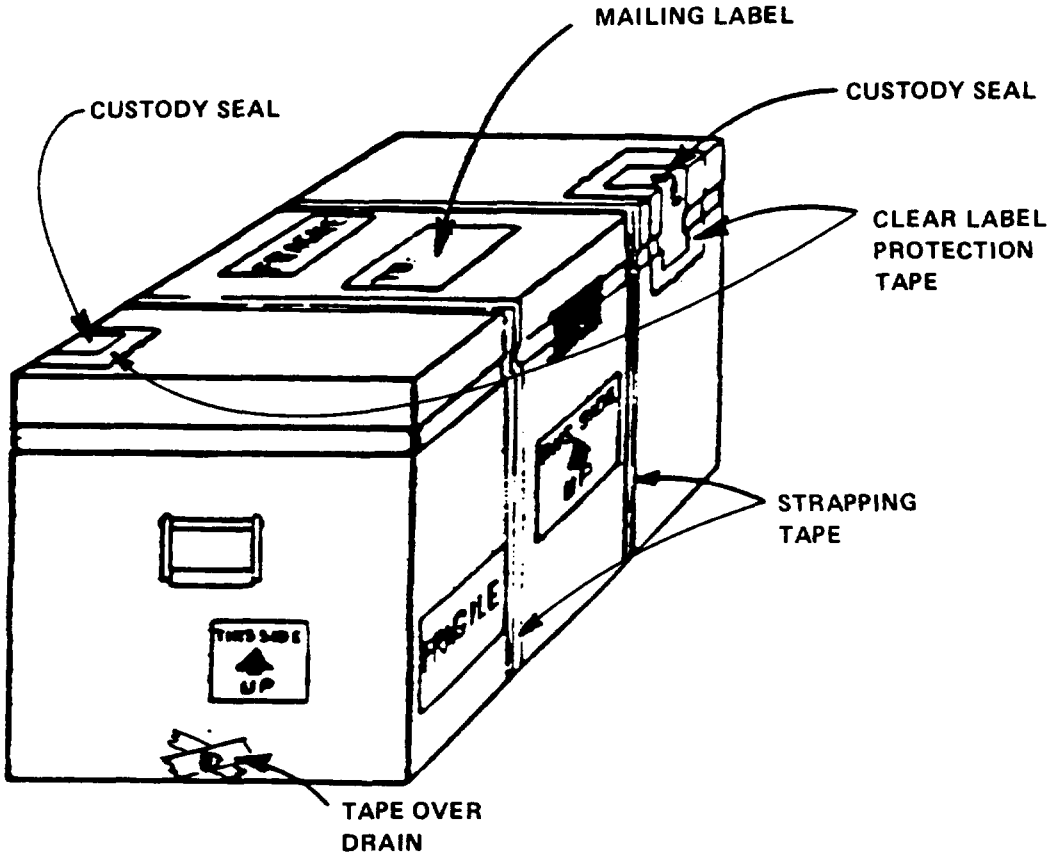

MAILING LABEL
CUSTODY SEAL
CUSTODY SEAL
CLEAR LABEL
PROTECTION
TAPE
STRAPPING
TAPE
THIS SIDE
UP
FRAGILE
TAPE OVER
DRAIN

FIGURE 10

Ms. Leslie Braun (subject to change)
(703) 557-2490

Provide the following information:

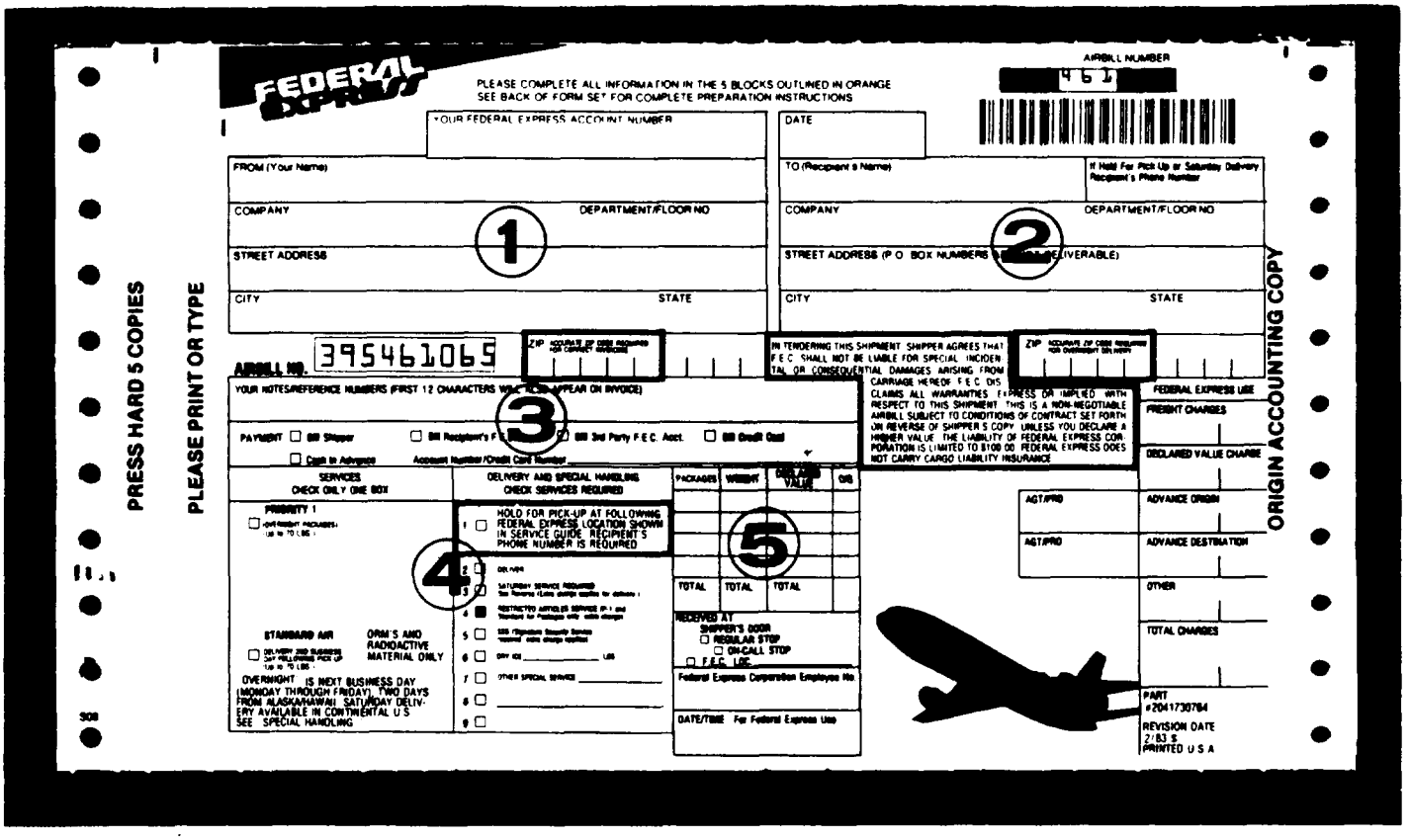
- Your name
- Project name
- Case number
- Number of samples sent to each laboratory for analysis
- Airbill numbers

This must be done immediately following sample shipment. (If the SMO is closed at that time, call in the information first thing the next day.)

MEDIUM- AND HIGH-CONCENTRATION SAMPLES

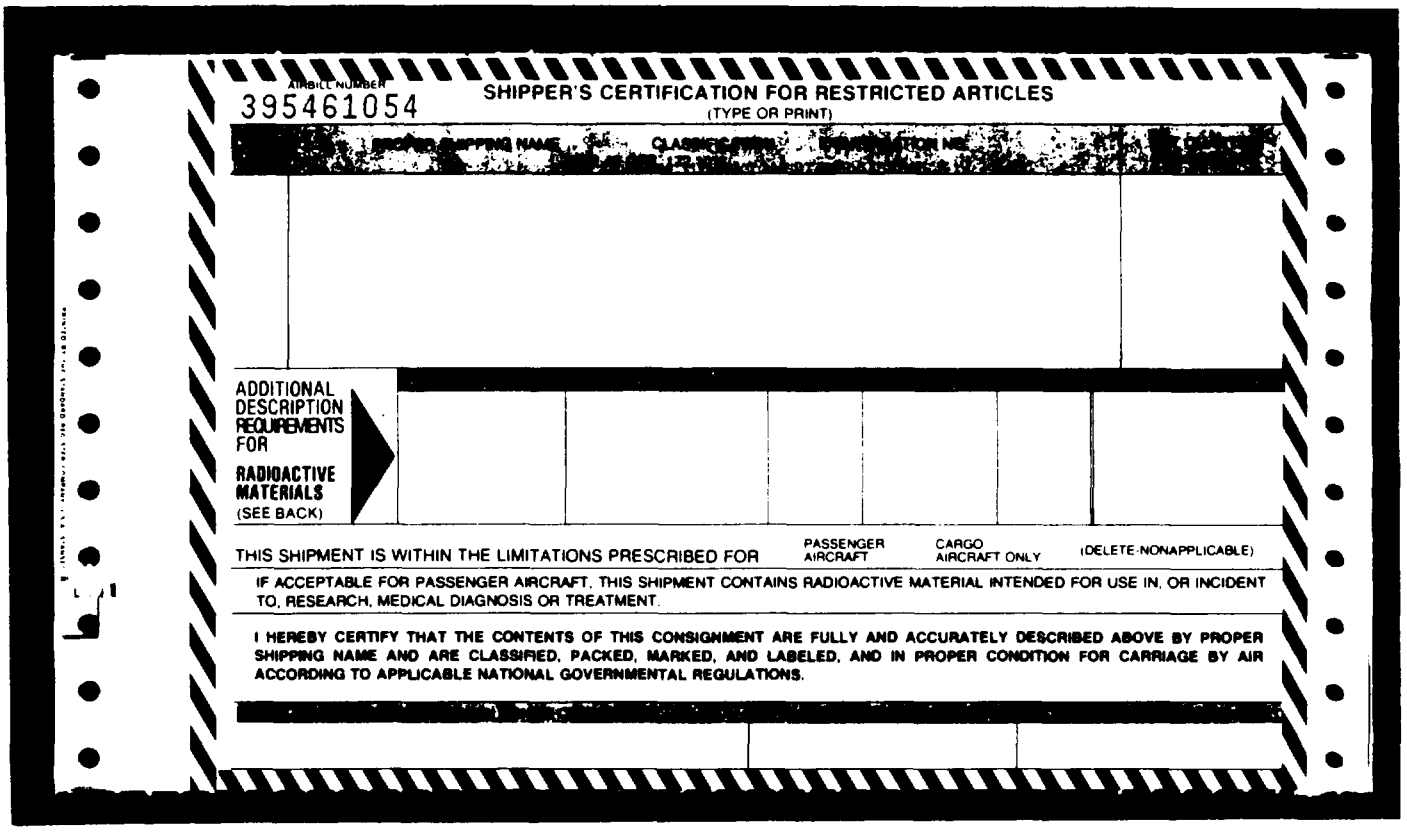
Medium- and high-concentration samples are packaged using the same techniques used to package low-concentration samples, with several additional restrictions. First, special airbill including a Shipper's Certification for Restricted Articles is required (see Figure 11 and 12). Second, "Flammable Liquid N.O.S." or "Flammable Solid N.O.S." labels must be placed on at least two sides of the cooler. Third, sample containers are packaged in metal cans with lids before being placed into the cooler, as indicated below.

- Place approximately one-half inch of vermiculite in the bottom of the can.
- Position the sample jar in the zip-loc bag so that the sample tags can be read through the plastic bag.
- Place the jar in the can and fill the remaining volume with vermiculite.
- Close the can and secure the lid with metal clips.
- Write the traffic report number on the lid.
- Place "This Side Up" and "Flammable Liquid N.O.S." (or "Flammable Solid N.O.S.") labels on the can.
- Place the cans in the cooler.



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 11



NOTE: For purposes of illustration forms are reproduced at 70% of original size.

FIGURE 12

DISTRIBUTION OF COMPLETED DOCUMENTS

Final disposition of the completed documents is as follows:

- Shipped with Samples:
  - Chain-of-custody form, white original
  - Traffic report forms, white and yellow copies
  - SAS packing list, pink and gold copies
  - Sample tags
- Retained by RI Project Manager:
  - Sample identification matrix
  - Field log books (at completion of project)
- Sent to CH2M HILL Documentation Coordinator:
  - Chain-of-custody form, pink and yellow copies
  - Traffic report forms, white original and pink copy
  - SAS packing list, white original and yellow copy
  - Notice of transmittal

SPECIAL INSTRUCTIONS FOR SHIPPING SAMPLES VIA FEDERAL EXPRESS

1. Label cooler as hazardous shipment.
   - Write shipper's address on outside of cooler. If address is stenciled on, just write "shipper" above it.
   - Write or affix sticker saying "This Side Up" on two adjacent sides.
   - Write or affix sticker saying "ORM-E" with box around it on two adjacent sides. Below ORM-E, write NA No. 9188.
   - Label cooler with "Hazardous Substance, NOS.", and "liquid" or "solid", as applicable.
2. Complete the special shipping bill for restricted articles (Figures 11 and 12).

- Under Proper Shipping Name, write "Hazardous Substance, NOS." and "liquid" or "solid", as applicable.
- Under Class, write "ORM-E."
- Under Identification No., write NA No. 9188.

GLT718/8